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16. ABSTRACT

The report gives currently available chemical data from 19 Level 1 environmental assessment studies, compiled in standard format. The data are organized within each study by the analytical technique used to generate them. Inorganic data generated by spark source mass spectroscopy (SSMS), atomic absorption (AA), gas chromatography (GC), chemiluminescence for oxides of nitrogen, anion analysis, and aqueous analysis precede organic data generated by GC for C1-C6/C7 or C7-C17, liquid chromatographic (LC) fractionation, infrared (IR) spectroscopy, and low resolution mass spectroscopy (LRMS). Each study is summarized, followed by the data generated in that study. The studies are organized by industrial type: chemically active fluidized bed combustor, coal-fired boiler, coal-fired power plant, new energy source, coke production, electric arc furnace, fluidized bed combustor, home heater multi-source source, ocean incinerator, oil burner, and textile. The report documents sampling and analytical techniques that were used which are not specified in Level 1. It also includes trends and anomalies that were detected in the 19 studies.

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by

N. H. Gaskins and F. W. Sexton

Research Triangle Institute
PO Box 12194
Research Triangle Park, North Carolina 27709

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Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
Research Triangle Park, NC 27711

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ABSTRACT

Currently available chemical data from 19 Level 1 environmental assessment studies are compiled in this document in standard formats. The formatted data are organized within each study by the analytical technique used to generate the data. Inorganic data as generated by Spark Source Mass Spectroscopy (SSMS), Atomic Absorption (AA), Gas Chromatography (GC), Chemiluminescence for oxides of nitrogen, Anion Analysis, and Aqueous Analysis precede the organic data generated by Gas Chromatography for C_1-C_6/C_7 or C_7-C_{17}, Liquid Chromatographic Fractionation (LC), Infrared Spectroscopy (IR), and Low Resolution Mass Spectroscopy (LRMS).

Each study is introduced by a summary which is then followed by the data generated in that study. The studies are organized by industrial type as follows: Chemically Active Fluidized Bed Combustor, Coal-Fired Boiler, Coal-Fired Power Plant, New Energy Source, Coke Production, Electric Arc Furnace, Fluidized Bed Combustor, Home Heater, Multi-Type Source, Ocean Incinerator, Oil Burner, and Textile.

Sampling and analytical techniques that were used which are not specified in Level 1 are documented in the summaries and data pages. This document also includes a section detailing some trends and anomalies that were detected among the 19 studies.

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IN BLACK AND WHITE

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LIST OF ABBREVIATIONS AND SYMBOLS

ABBREVIATIONS

AA or AAS	-- atomic absorption spectroscopy
ADL	-- Arthur D. Little, Inc.
AN	-- Army-Navy (Swagelok-type fittings)
ANL	-- Argonne National Laboratory
APS	-- ammonium persulfate
ASTM	-- American Society for Testing and Materials
ATMI	-- American Textile Manufacturers Institute
B(a)P	-- benzo(a)pyrene
BATEA	-- best available technology, economically achievable
BCL	-- Battelle, Columbus Laboratories (Ohio)
BOD_5	-- five-day biological oxygen demand test
CAFB	-- chemically active fluidized bed
COD	-- chemical oxygen demand test
dscm	-- dry standard cubic meters
EC	-- electron capture detection, for gas chromatography
EM	-- electron microprobe
ESCA	-- electron spectroscopy for chemical analysis
ESP	-- electrostatic precipitator
FBC	-- fluidized-bed combustor
FID	-- flame ionization detection, for gas chromatography
FPD	-- flame photometric detection, for gas chromatography
FTIR	-- Fourier Transform infrared spectroscopy
GC	-- gas chromatography
GCA	-- Geophysical Corporation of America
GPC	-- gel permeation chromatography
GRAV	-- determinations of nonvolatile or gravimetric solids
HC	-- hydrocarbon
HGA	-- hollow graphite atomizer
HPLC	-- high performance liquid chromatography
HVSS	-- high-volume sampling system
IC	-- ion chromatography
ICAP	-- inductively coupled argon plasma
IERL	-- Industrial Environmental Research Laboratory of EPA
IR	-- infrared spectroscopy
K-D	-- Kuderna-Danish apparatus
LC	-- liquid chromatography
LLL	-- Lawrence Livermore Laboratories
LRMS	-- low-resolution mass spectroscopy
MATE	-- minimum acute toxicity [value for the] effluent
MDL	-- minimum detection limit
MERC	-- Morgantown Energy Research Center of the Energy Research and Development Agency

MMEG	-- Multimedia Environmental Goals
MRC	-- Monsanto Research Corporation
MS	-- mass spectrometry
MW	-- megawatt
NBS	-- National Bureau of Standards
NDIR	-- nondispersive infrared spectroscopy
OES	-- optical emission spectroscopy
ORNL	-- Oak Ridge National Laboratory
PCB	-- polychlorinated biphenyls
P&E	-- Perkin-Elmer Company
PFBC	-- pressurized fluidized-bed combustor
PMB	-- The Process Measurements Branch of the IERL
PNA	-- polynuclear aromatics
POM	-- polycyclic organic material
ppm	-- parts per million, by volume
ppmw	-- parts per million, by weight
RAC	-- Research Appliance Corporation
SASS	-- source assessment sampling system
SDDC	-- silver diethyl dithio carbamate
SRC	-- solvent-refined coal
SRM	-- Standard Reference Material, issued by the National Bureau of Standards
SS	-- stainless steel
SSMS	-- spark source mass spectroscopy
TCO	-- total chromatographable organics (volatiles)
TGA	-- thermo-gravimetric analysis
TLC	-- thin layer chromatography
TSS	-- total suspended solids
TVA	-- Tennessee Valley Authority
UV	-- ultraviolet wavelengths
VIS	-- visible light wavelengths

SYMBOL

Nm^3	-- normal cubic meters (at standard temperature and pressure)
μ	-- micrometer (also written μm)

INTRODUCTION

This document is an accumulation of all available chemical data through February 1978 from Level 1 of the phased environmental assessment program. The primary purpose of this compilation is to permit those involved in environmental assessment programs to evaluate the quality and quantity of data generated by the phased approach. It is felt that critical reviews of these data may lead to improvements in procedures, data formatting, data storage, and interpretation. Although conclusions related to specific sources or source types may have been abstracted from the references to provide background information, the focus of this presentation is on data resulting from the sampling and analytical methods. The interested reader should consult the referenced documents for more details and conclusions concerning pollutant sources, control, etc.

The phased environmental assessment program, developed by the Industrial Environmental Research Laboratory (IERL) of the Environmental Protection Agency (EPA) at Research Triangle Park (RTP), North Carolina, is structured in three phases. Level 1 is the survey step to determine which samples from an environmental assessment might be hazardous or toxic. Level 1 also serves to prioritize and rank samples for further testing. When the Level 1 sampling and analysis scheme shows the possible presence of hazards, a Level 2 scheme is initiated to specifically identify and quantify suspected hazardous materials. If Level 2 reveals pollutants capable of environmental detriment then a Level 3 scheme is begun to evaluate control technologies and to assess long-term effects.

At the present time, Levels 2 and 3 are in the developmental stages. Nineteen studies have been identified that contain Level 1 data, and these studies are organized alphabetically by source types in this compilation. It should be noted that each study included in this compilation will be identified as final draft, preliminary draft, etc., based on the completeness of the study summarized; therefore, absence of data does not necessarily indicate analysis was not performed but that the study was possibly not complete when it became necessary to compile the data. In each study, mention is made of methods used if they differ from the methodology specified in the IERL-RTP Procedures Manual: Level 1 Environmental Assessment.

Within each study, information is organized in the following manner:

I. Summary of the study

    A. General information, purpose, conclusions
    B. Gaseous grab samples--sampling and analysis
    C. Source Assessment Sampling System (SASS) samples--sampling and analysis
    D. Fugitive emissions--sampling and analysis

   - E. Liquids and slurries--sampling and analysis
   - F. Solids--sampling and analysis

II. Formatted data

   - A. Inorganic analysis data
      1. Spark Source Mass Spectroscopy (SSMS)
      2. Atomic Absorption (AA) wet chemical methods--Hg, As, Sb
      3. Gas Chromatography for inorganic gases
      4. Chemiluminescence for NO_x
      5. Anion analysis
      6. Aqueous analyses for pH, acidity, alkalinity, BOD, COD, DO, conductivity, dissolved and suspended solids, species analysis (anions)
   - B. Organic analysis data
      1. Gas Chromatography for C_1-C_6/C_7
      2. Gas Chromatography for C_7-C_{17}
      3. LC fractionation
      4. IR report
      5. Low Resolution Mass Spectrometry (LRMS)

The first section of this document contains a collective evaluation of data from all of the nineteen formatted environmental assessment studies. This evaluation was directed at identifying trends or anomalies that were located during data compilation. The primary focus of this effort was to compose the data among the different industrial sources in an effort to characterize the Level 1 analytical techniques.

The following section contains this evaluation of trends and anomalies in the compiled data which is in turn followed by the compiled data from each study.

TRENDS AND ANOMALIES IN DATA

A brief assessment of trends and anomalies in data from the nineteen environmental assessment studies was made. Topics addressed in this section include the following:

1. Particulate size vs. selective trace element concentrations;
2. Contaminants in LC fractions;
3. Aliphatic hydrocarbon presence in LC fractions;
4. Aromatic hydrocarbon presence in LC fractions;
5. LC fractions containing similar structures;
6. Analytical techniques used in Level 1 environmental assessments; and
7. Organic concentration distributions from two different LC fractionation schemes.

It should be understood that the data generated during these environmental assessments are sample- and source-dependent. In addition, there was a limited number of studies available for trend evaluation, and the analytical methodologies used varied with some studies. The conclusions to follow were made under these constraints and should be interpreted accordingly.

Conclusions from these evaluations follow:

1. In more than 55 percent of the samples analyzed by Spark Source Mass Spectroscopy, Be, Mo, Zn, Hg, As, and Sb concentrated in particulates less than 3μ in size. In more than 55 percent of the samples, Sr, Cu, and Ga concentrated in particulates more than 3μ in size. Pb, Se, and Cd did not show a preferential concentration with size (i.e., 50 percent ± 5 percent of samples concentrated in either particulate size range).

2. Contaminants such as silicone compounds and phthalates exhibit no preference as to the LC fraction in which they are found, while water tends to elute in fractions 6, 7, or 8.

3. Of the 78 samples that elute aliphatic hydrocarbons in fraction 1, 29 samples (37 percent) also showed the presence of aliphatic hydrocarbons in fraction 2. The presence of these aliphatics in fractions other than 1 or 2 accounts for less than 1 percent of the samples tested. Samples from studies 16 and 19 were interpreted by the investigators to contain such a high aliphatic HC content that they tended to bleed across all eight fractions.

4. Aromatic structures were frequently not fully identified as being aromatic hydrocarbon or other chemical structure. Of the 24 samples containing aromatic hydrocarbons in fraction 2, 10 samples (41 percent) showed aromatic hydrocarbons in fraction 3 while 16 samples (67 percent) showed aromatic hydrocarbons in fraction 1.

5. IR data indicate that fractions 3 and 4 contained identical structures more frequently than any other combination of adjacent fractions.

6. Analytical techniques used most frequently in effluent characterization studies were SSMS, LC fractionation, and LRMS. Aqueous analyses and field analyses for C_1-C_6 gases were seldom used.

7. Evaluation of the gravimetric data indicates that in the Level 1 liquid chromatographic fractionation, the lowest amount of organics is found in fractions 2 and 5. This is determined by evaluation of data from six different industrial sources.

DATA AND DISCUSSION

Trace Elemental Concentration

The trace elements Pb, Se, Cd, Cu, Ga, Mo, Zn, Hg, As, and Sb were selected for comparison of concentration variations with particulate size because of the high concentrations of these elements in fly ash as previously determined by ORNL.[1] Be and Sr were also included for comparison due to potential problems that may result from their presence in larger size particulate.[2]

SSMS data on particulates from 14 different samples were compared to determine which size particulate contained the highest concentration. These data are summarized in Table 1. The percentage of samples in which particulates less than 3μ contained the higher concentration is presented in Figure 1.

LC Fractionation--Contaminants and Hydrocarbons

Interpretations of IR data from each study were inspected to determine what fractions repeatedly contained suspected contaminants (silicone compounds, water, phthalates) and also to determine where aliphatic and aromatic hydrocarbons eluted. These data are summarized in Table 2.

Similarities of LC Fractions

The interpretations of IR data in the formatted studies were generally presented in text form. These IR interpretations were inspected to determine which LC fractions contained identical structures. Those fractions containing the same structures are so designated by use of the same symbol in Table 3. The frequency with which adjacent LC fractions contain identical structures is shown in Table 4.

---

[1] Klein, David H., et al. Pathways of Thirty-seven Trace Elements Through a Coal-Fired Power Plant. Environmental Science and Technology, 9 (10):973-979, October 1975.

[2] Dorsey, James A. Process Measurements for IERL/RTP Environmental Assessment Programs. Environmental Protection Agency, Process Measurements Branch, August 1977.

Use of Analytical Techniques

Table 5 identifies the analytical techniques that were used in each study. Incomplete studies are identified.

Organic Distribution by Fraction

Table 6 reports the percentage of organics separated by each fraction of an LC fractionation scheme. The percentages for each sample are determined by the following equation:

$$\frac{\text{Fraction weight}}{\text{Total of fraction weights}} \times 100 = \text{Fractional percentage.}$$

These results are presented by source type and fractionation method used: the BCL method as reported in Technical Manual for Analysis of Organic Materials in Process Streams or the original Level 1 method as reported in the Environmental Assessments Procedures Manual. The solvents and volumes used in these methods follow:

BCL

Fraction	Volume (ml)	Solvent
1	25	60/80 pet ether
2	25	20% CH_2Cl_2/pet ether
3	25	50% CH_2Cl_2/pet ether
4	25	CH_2Cl_2
5	25	5% CH_3OH/CH_2Cl_2
6	25	20% CH_3OH/CH_2Cl_2
7	25	50% CH_3OH/CH_2Cl_2
8	25	CH_3OH

Original Level 1

Fraction	Volume (ml)	Solvent
1	25	Pentane
2	10	20% CH_2Cl_2/Pentane
3	10	50% CH_2Cl_2/Pentane
4	10	Same as BCL
5	10	Same as BCL
6	10	Same as BCL
7	10	Same as BCL
8	10	5/70/30, Conc. $HCl/CH_3OH/CH_2Cl_2$

(Revised Level 1 eliminates Fraction 8.)

TABLE 1. DISTRIBUTION OF HIGHEST TRACE ELEMENT CONCENTRATION BY PARTICULATE SIZE CATEGORY*

Study no.		Be	Sr	Pb	Se	Cd	Cu	Ga	Mo	Zn	Hg	As	Sb
3	Run 1	‡	‡	‡	>3	>3	>3	<3	<3	<3	<3	<3	<3
10	Run 2	>3	‡	†	†	†	†	†	†	†	>3	<3	<3
	4	†	†	†	†	>3	>3	>3	>3	†	<3	<3	<3
	5	<3	†	<3	<3	†	<3	>3	<3	<3	†	<3	<3
11	Run 2	<4.6	>4.6	>4.6	>4.6	>4.6	>4.6	>4.6	>4.6	<4.6	>4.6	<4.6	>4.6
	3	<4.6	<4.6	>4.6	>4.6	>4.6	>4.6	>4.6	<4.6	<4.6	<4.6	<4.6	>4.6
	4	†	†	>4.6	>4.6	†	†	<4.6	<4.6	>4.6	<4.6	<4.6	<4.6
15	Source 1	<3	<3	<3	<3	<3	>3	>3	<3	†	NR	<3	<3
	3	>3	>3	>3	>3	<3	>3	>3	>3	‡	NR	>3	>3
	4	>3	>3	<3	<3	<3	<3	>3	<3	<3	NR	<3	<3
	6	<3	>3	>3	‡	‡	>3	>3	‡	<3	NR	>3	<3
	7	<3	>3	<3	<3	<3	>3	<3	<3	<3	NR	<3	<3
	8	<3	>3	<3	<3	<3	<3	<3	<3	<3	NR	<3	<3
	9	>3	>3	>3	<3	>3	†	<3	<3	†	NR	<3	>3
Total <3μ		7	2	5	6	5	3	5	9	8	4	12	10
Total Samples		11	9	11	11	10	11	13	12	9	6	14	14
Percent <3μ		63.6	22.2	45.5	54.5	50	27.3	38.5	75	88.9	66.6	85.7	71.4

*Particulate size categories are reported in microns.
†Concentration did not vary more than 10 percent with particulate size.
‡Insufficient data.

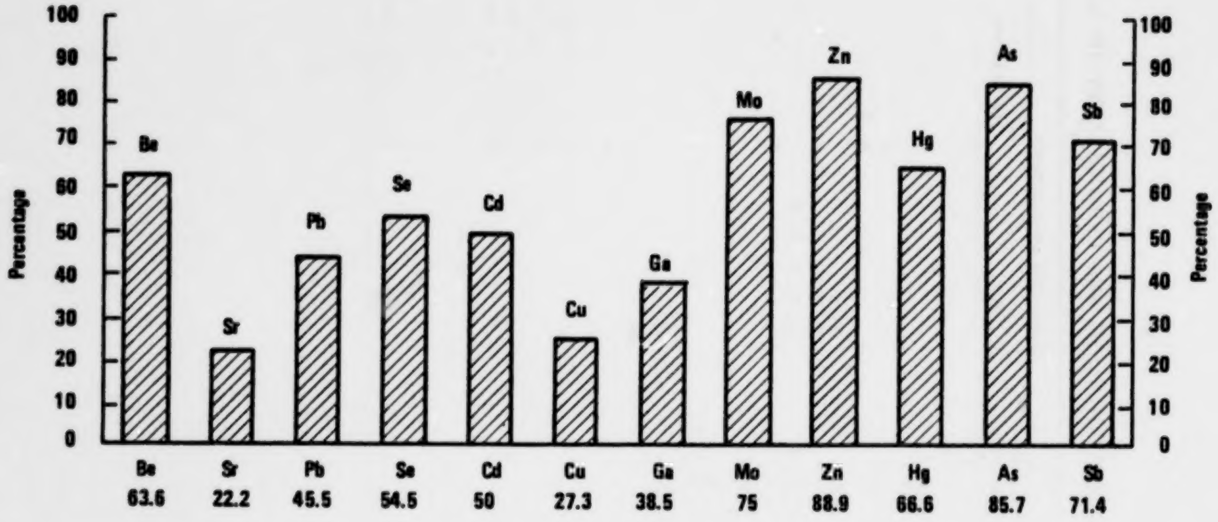

Figure 1. Percent of samples in which elements concentrated in particulates $< 3\mu$.

TABLE 2. PRESENCE OF CONTAMINANTS AND HYDROCARBONS IN LC FRACTIONS AS DETERMINED BY IR

Study no.	Table no.	Sample	Suspected contaminants			Hydrocarbons		Fractionation scheme†
			Silicone compounds*	H_2O	Phthalates	Aliphatic	Aromatic	
1	9	Coal	6, 7, 8				2‡	BCL
	10	Flue gas	1, 8		8	1, 2, 7		
	11	Particulates	4, 8			1		
	12	Particulates			4	1	4‡	
	13	Bed material			6, 8	1, 2, 3, 4, 5		
3	15	Module wash	8	7, 8				Level 1
	16	Cannister wash		7, 8				
	17	Coal	1, 2, 4	4, 5, 6, 7, 8		1, 2	2‡, 4‡, 5‡	
4	4-J1	Coal	1, 2, 3		6, 7		1, 2, 3	BCL
	4-J2	Coal	1, 2, 3				1, 2	
	5	Coal	1					
	6-J3	Coal	1, 2				2	
	6-J4	Coal	1, 2				2	
5	5	Multi		8?		1		Level 1
6	7	Sample 2		7, 8		1		Level 1
	7	Sample 3				1		
	7	Sample 5				5	5‡	
7	8	Test 13	1		2, 3, 4, 5, 6#	1, 2	1	BCL
	9	Clean H_2O	1, 2		2, 3, 4, 5, 6, 7#	1		
	10	Blank	1		3, 4, 5, 6#	1, 2	2	
	11	Coke--clean			3, 4, 5, 6, 7#	1		
	12	Coke--dirty	1, 2		3, 4, 5, 6, 7#	1, 2		
	13	Dirty H_2O				1	1, 2	
	14	Liquor				1		
	15	Clean H_2O				1		
	16	Dirty H_2O				1		
	17	Test 16	1, 2		2, 3, 4, 5, 6#	1		

See footnotes at end of table.

(continued)

TABLE 2 (continued)

Study no.	Table no.	Sample	Suspected contaminants			Hydrocarbons		Fractionation scheme†
			Silicone compounds*	H_2O	Phthalates	Aliphatic	Aromatic	
8	10	Adsorber - A1				1, 2	2, 3	BCL
		A2				1, 2	1, 2	
		A3				1, 2	1, 2	
	11	A4				1, 2	1, 2	
		A5				1, 2	1, 2	
		Air A6	2		7	1, 2	3†, 4†	
	12	Filter A1F				1, 2	1, 2, 3	
		A2F				1, 2	1, 2, 3	
	13	A3F				1, 2	1, 2, 3	
		A4F				1, 2	1, 2, 3	
		A5F			5, 6, 7	1, 2	1, 2, 3	
10	77	Coal - Run 2		8		1, 2	2, 3, 4	Level 1
		Run 5		8		1, 2, 4	2, 3	
	78	Flue gas - Run 1	2, 3, 4, 5	8		1		
		2	2, 3, 4, 5, 6			1		
		3				1		
		4				1		
		5	2, 3			1		
	79	Air - Run 1		7, 8		1		
		2		7		1		
	80	Particulates >3μ						
		Run 2	1, 6			1		
		4						
		5		1		1		
		Particulates <3μ						
		Run 2		4		1	5	
		4		1		1		
		5			8	1		
	81	SASS - Run 2	3, 4, 5	7				
		4		8	5	2		
		5		8	4, 5, 6, 8	1		

See footnotes at end of table.

(continued)

TABLE 2 (continued)

Study no.	Table no.	Sample	Suspected contaminants			Hydrocarbons		Fractionation scheme†
			Silicone compounds*	H_2O	Phthalates	Aliphatic	Aromatic	
	82	Bed reject - 2						
		4		7, 8	1, 2	1		
		5		8	2?, 3?	1		
	83	Ash - Run 2				1		
		4				1, 2, 3, 4, 7		
		5			8			
11	44	Coal Culm - Run 2						Level 1
		Run 3			5, 6			
		Run 4						
	45	Bed reject - Run 2				1		
		3				1		
		4			5, 6	1		
	46	Ash - Run 3			3	1		
		Run 4			4	1		
	47	Ash - Run 2				1		
		3						
		4			3	1		
	48	Tenax - Run 2	1, 2, 3				1, 2	
		3	1, 2			1		
		4	1, 2			1		
		Blank				1		
	49	Particulates						
		Run 2						
		3				1	1?	
		4				1, 2	2	
	50	Filter						
		Run 2				1		
		3				1	1	
		4				1		
12	24	Particulates -						Level 1
		>27μ			4			
		<27μ			4	1		

See footnotes at end of table.

(continued)

TABLE 2 (continued)

Study no.	Table no.	Sample	Suspected contaminants			Hydrocarbons		Fractionation scheme†
			Silicone Compounds*	H_2O	Phthalates	Aliphatic	Aromatic	
	25	Bed material				1		
		Ash			5	1, 2, 3		
	26	Sludge				1, 2	1, 2	
	27	Particulate -						
		>27μ				1		
		<27μ	1			1, 2	1, 2	
13	57	Site 300				1		Level 1
		301				1, 2		
		302						
		303				1		
		304			7	1, 2	1‡, 2‡	
14	IR DATA FROM THIS STUDY ARE NOT BLANK CORRECTED.							---
16	3	Coal	6, 7, 8			1, 2, 3, 4, 5, 6, 7, 8	2‡, 3‡	BCL
	4	Steam				1, 2, 3, 4, 5, 6, 7, 8		
	5	Multi					1, 2	
	6	Oil	1, 2, 7, 8	7, 8		2, 3, 4, 5		
18	11	Probe - Run 1	7, 8	8	6	1		Level 1
		2	7, 8	7, 8	6			
		Module - Run 1	1, 3, 4	7	4, 5, 6	1		
		2	3	8	5, 7	1, 2	2, 3, 4, 5‡	
19	35	Plant A	6, 7, 8	7, 8	6	Incomplete classification due to high hydrocarbon concentrations.		
	36	B	1, 8					
	37	E	8	2, 8				
	38	F	1, 6, 8					
	39	G	6, 7, 8					

See footnotes at end of table.

(continued)

TABLE 2 (continued)

Study no.	Table no.	Sample	Suspected contaminants: Silicone compounds*	Suspected contaminants: H_2O	Suspected contaminants: Phthalates	Hydrocarbons: Aliphatic	Hydrocarbons: Aromatic	Fractionation scheme†
	40	K	1, 2, 3, 4, 5 6, 7, 8	1		Incomplete classifications due to high hydrocarbon concentrations.		
	41	L	8	8				
	42	N						
	43	S	6?, 7?, 8					
	44	T	3, 4, 5, 6? 7?, 8?					
	45	U	8					
	46	V	2, 4, 5, 6, 8					
	47	W		5?, 8				
	48	X						

*To include silicon dioxide, silicon, and silica gel.

†Fractionation scheme:
BCL's method as presented in Technical Manual for Analysis of Organic Material in Process Streams.
Level 1 method as presented in the Environmental Assessments Procedures Manual.

‡Incomplete classification of aromatic structure.

#Authors of study number 7 indicated phthalates were probably actual constituents of the effluent stream rather than contaminants of the fractionation scheme.

TABLE 3. LC FRACTIONS CONTAINING IDENTICAL STRUCTURES AS DETERMINED BY IR

Study no.	Table no.	Sample	Fraction 1	2	3	4	5	6	7	8
1	9	Coal	*		*	*		†	†	†
	10	Flue gas	*	*	†	†			*	
	11	Particulates[a]								
	12	Particulates				*	*			
	13	Bed material	*	*		†	†	‡		‡
3	15	Module wash[a]								
	16	Cannister wash[a]								
	17	Coal						*	*	
4	4-J1	Coal	*	*						
	4-J2	Coal[a]								
	5-	Coal		*	*	†	†			
	6-J3	Coal			*	*				
	6-J4	Coal			*	*				
5	5	Multi[a]								
6	7	Sample 2		*	*					
	7	Sample 3[a]								
	7	Sample 5			*	*				
7	8	Test 13			*	*	†	†		
	9	Clean H_2O			*	*				
	10	Blank							*	*
	11	Coke--clean					*	*		
	12	Coke--dirty			*	*				
	13	Dirty H_2O					*	*		
	14	Liquor				*	*	*		
	15	Clean H_2O[a]								
	16	Dirty H_2O[a]								
	17	Test 16			*	*	†	†		
8	10	Adsorber - A1						*	*	
		A2				*	*	†	†	
		A3				*	*	†	†	

[a] No fractions contain identical structures.

(continued)

TABLE 3 (continued)

Study no.	Table no.	Sample	Fraction 1	2	3	4	5	6	7	8
	11	A4						†	†	
		A5						†	†	
		Air A6			*	*				
	12	Filter A1F	*	*						
		A2F	*	*			†	†		
	13	A3F	*	*				†	†	
		A4F	*	*						
		A5F	*	*						
10	77	Coal - Run 2[a]								
		Run 5[a]								
	78	Flue gas - Run 1		*	*					
		2		*		*	*			
		3[a]								
		4[a]								
		5[a]								
	79	Air - Run 1[a]								
		2[a]								
	80	Particulates >3μ								
		Run 2[a]								
		4[a]								
		5[a]								
		Particulates <3μ								
		Run 2[a]								
		4							*	*
		5[a]								
	81	SASS - Run 2			*	*				
		4[a]								
		5[a]								
	82	Bed reject								
		Run 2[a]								
		4[a]								
		5		*	*					

---

[a] No fractions contain identical structures.

(continued)

TABLE 3 (continued)

Study no.	Table no.	Sample	Fraction 1	2	3	4	5	6	7	8
	83	Ash - Run 2[a]								
		4	*	*	*					
		5		*	*	*	*			
11	44	Coal culm								
		Run 2[a]								
		3[a]								
		4[a]								
	45	Bed reject								
		Run 2[a]								
		3[a]								
		4					*	*		
	46	Ash - Run 3[a]								
		4[a]								
	47	Ash - Run 2				*	*	*		
		3[a]								
		4[a]								
	48	Tenax - Run 2					*	*		
		3			*	*	†	†	‡	‡
		4			*	*	*			
		Blank				*	*		†	†
	49	Particulates								
		Run 2		*	*	†	†	†		†?
		3			*	*	*			
		4				*	*			
	50	Filter - Run 2		*	*					
		3		*	*					
		4[a]								
12	24	Run 1								
		Particulates >27μ[a]								
		<27μ[a]								
	25	Bed material		*	*	*	*	*		
		Ash[a]								

---

[a] No fractions contain identical structures.

(continued)

TABLE 3 (continued)

Study no.	Table no.	Sample	Fraction 1	2	3	4	5	6	7	8
	26	Sludge						*	*	
	27	Run 2								
		Particulates >27μ					*	*	*	
		<27μ[a]								
13	57	Site 300							*	*
		301			*	*	*			
		302[a]								
		303			*	*	†	†		
		304			*	*	†	†		
16	3	Coal	†	*	*	†		‡	‡	‡
	4	Steam		*	*	*		*	†	†
	5	Multi[a]								
	6	Oil			*	*			†	†
18	11	Probe - Run 1[a]								
		2[a]								
		Module - Run 1[a]								
		2[a]								
19	35	Plant A			*	*				
	36	B[a]								
	37	E[a]								
	38	F						*	*	
	39	G				*	*	†	†	
	40	K						*	*	
	41	L[a]								
	42	N[a]								
	43	S				*	*			
	44	T			*		*			
	45	U[a]								
	46	V				*	*			
	47	W[a]								
	48	X			*	*		†	†	

[a] No fractions contain identical structures.

TABLE 4. PERCENTAGE OF LC FRACTIONS CONTAINING IDENTICAL STRUCTURES AS DETERMINED BY IR

	BCL fractionation scheme*						
Fractions identical	1≡2	2≡3	3≡4	4≡5	5≡6	6≡7	7≡8
Occurrences identical	8	3	11	6	6	8	4
Total samples	35	35	35	35	35	35	35
Percent (%)‡	22.9	8.6	31.4	17.1	17.1	22.9	11.4
Ranking	2	5	1	3	3	2	4
	Original Level 1 fractionation scheme†						
Fraction identical	1≡2	2≡3	3≡4	4≡5	5≡6	6≡7	7≡8
Occurrences identical	1	9	12	13	9	7	4
Total samples	82	82	82	82	82	82	82
Percent (%)‡	1.2	11.0	14.6	15.9	11.0	8.5	4.9
Ranking	6	3	2	1	3	4	5
	Summation of both fractionation schemes						
Fractions identical	1≡2	2≡3	3≡4	4≡5	5≡6	6≡7	7≡8
Occurrences identical	9	12	23	19	15	15	8
Total samples	117	117	117	117	117	117	117
Percent (%)‡	7.7	10.3	20.0	16.2	12.8	12.8	6.8
Ranking	5	4	1	2	3	3	6

*Used in Studies 1, 4, 7, 8, and 16.

†Used in Studies 3, 6, 10, 11, 12, 13, 18, and 19.

‡Percent = $\frac{\text{occurrences identical}}{\text{total samples}} \times 100$.

TABLE 5. USE OF ANALYTICAL TECHNIQUES

Study no.	SSMS	AA	GC	Chemiluminescence (NO_x)	ANION	AQUEOUS	C_1-C_6	C_7-C_{17}	LC	IR	LRMS
1	X								X	X	
2*			X	X			X				
3	X	X			X				X	X	X
4									X	X	
5*			X		X				X	X	
6*	X	X						X	X	X	
7	X								X	X	
8	X								X	X	
9									X	X	
10*	X	X	X	X	X			X	X	X	X
11*	X	X	X	X	X				X	X	
12	X	X	X	X	X				X	X	
13*	X	X	X				X	X	X		
14*									X	X	X
15	X										
16									X	X	
18*	X	X						X	X	X	X
19	X	X				X			X	X	
Total	12	8	6	4	5	1	2	4	16	15	4

*These (draft) reports may not contain results from all analyses.

TABLE 6. PERCENTAGE OF ORGANIC DISTRIBUTION BY LC FRACTION

Fractionation method	Study	Source type	LC Fraction 1	2	3	4	5	6	7	8
			%	%	%	%	%	%	%	%
BCL	1	CAFB	28	8	9	14	17	14	3	7
BCL	4	Coal-fired plant	5	8	2	22	35	25	2	1
BCL	7	Coke production	12	9	26	9	19	3	18	4
BCL	8	Coke production	24	33	14	8	7	3	10	1
BCL	16	Multisource	31	3	3	21	21	7	10	4
BCL Average % Organics			20	12.2	10.8	14.8	19.8	10.4	8.6	3.4
Ranking			1	4	5	3	2	6	7	8
Level 1	3	Coal-fired boiler	3	2	8	2	1	7	26	51
Level 1	10	FBC	9	8	10	10	10	20	15	18
Level 1	11	FBC	34	5	13	8	2	10	22	6
Level 1	12	FBC	43	8	12	9	5	8	13	2
Level 1	13	Home heaters	15	4	6	22	11	22	20	0
Level 1	18	Oil burner	3	0.4	1	1	0.6	6	38	50
Level 1	19	Textile	13	8	12	5	3	24	16	19
Level 1	6	New energy	12	3	6	3	5	55	6	10
Level 1 Average % Organics			16.5	4.8	8.5	7.5	4.7	19	19.5	19.5
Ranking			4	7	5	6	8	3	1	2

STUDY NUMBER 1

DATA SOURCE:	**PRELIMINARY ENVIRONMENTAL ASSESSMENT OF THE CAFB** EPA-600/7-76-017
DATA STATUS:	Final Report, October 1976
AUTHORS:	Arthur S. Werner, Charles W. Young, Mark I. Bornstein, Robert M. Bradway, Michael T. Mills, and Donald F. Durocher
CONTRACTOR:	GCA Corporation GCA/Technology Division Bedford, Massachusetts 01730 Contract No. 68-02-1316, Task 14 Program Element No. EHB537
TASK OFFICER:	Samuel L. Rakes Industrial Environmental Research Laboratory U.S. Environmental Protection Agency Office of Energy, Minerals, and Industry Research Triangle Park, NC 27711

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GENERAL

"The Chemically Active Fluidized Bed (CAFB) is a technique whereby high sulfur, high metal residual oil is vaporized in a fluidized bed of lime to produce a low Btu, low sulfur product gas which is then burned in a conventional boiler to generate electrical energy. Most of the sulfur and metals contained in the oil feed are captured by the lime. This spent lime is subsequently processed to recover sulfur."* The process flow is diagrammed in Figure 1 from the study with mass flow rates given in Table 1.

A preliminary environmental assessment of the 2.93-MW CAFB process at the Esso Research Centre, Abingdon, England, was performed, and results of this study were used to predict environmental impact of the Foster Wheeler 10-MW retrofit demonstration plant to be constructed at La Palma Power Station in San Benito, Texas, and a conceptual design for a 250-MW commercial scale unit. Very thorough discussions of operating parameters were included. Appendixes discussed other sulfur removal technologies.

The process-pollutants released to the air from the boiler stack or from fugitive emissions of feed materials were the primary concern of this study. Water effluents were characterized as being similar to those of a conventional coal combustion plant (boiler blowdown, cooling tower outputs). The land pollutant of primary concern is the spent, sulfided limestone.

Seven separate analytical runs were made: four fuel oil gasification runs, two bitumen gasification runs, and one combustion/startup bitumen run. (See Table 2, Summary of Sampling Activity, from the document.) Results of analyses were used in dispersion modeling analysis to predict impact of the 10-MW plant on the San Benito area. A final section thoroughly discussed alternative residual oil utilization techniques and environmental considerations as well as capital and operating costs.

The study concluded that top priority environmental problems of CAFB operation include reduction of stack particulate emissions, reduction of SO_2 emissions during abnormal operating conditions, and disposal of spent lime. A need for further study of organic stack emissions (possible presence of MMEG levels of quinone, carbonyl compounds, and aliphatic hydrocarbons), for measurement of fugitive emissions from oil storage and bitumen storage and handling (bitumen is used in the RESOX^{TM} process, which reduces SO_2 effluent from the lime regenerator to elemental S), and for acceptable disposal methods for the RESOX^{TM} coal ash were indicated.

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*Preliminary Assessment of the CAFB, Werner, Young, and Bornstein.

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GASEOUS GRAB

The Level 1 glass bulb sampler was not used. Flue gases were sampled for NO_x by Federal Register Method 7, for SO_2/SO_3 by Method 8, for H_2S by Method 11, and for CO, CO_2, and O_2 by Orsat.

SASS

A SASS train was not used in this study. Particulate sampling was done with a standard RAC train using Method 5. Particle size distribution measurements were made with a University of Washington eight-stage cascade impactor using ungreased substrates. Organics were adsorbed on a Tenax GC gas adsorber cell, which was attached to the RAC train. After 1-hour sampling periods, adsorber cells were capped and stored in darkness until analysis. Pentane was used to extract organic vapors adsorbed on the Tenax polymer.

FUGITIVE EMISSIONS

Sampling for fugitive emissions was not performed. Mathematical modeling from fuel oil, RESOX™ coal, and limestone analyses gave estimates of fugitive emissions from the storage and handling of these materials.

LIQUIDS AND SLURRIES

Fuel oil was analyzed for organics (LC-IR) and for trace elements (SSMS-AA-wet chem). Specific sampling methods were not presented.

SOLIDS

Sampling methods for solids were not detailed. The following materials were analyzed: bitumen (organics), limestone, (surface composition by electron spectroscopy for chemical analysis [ESCA]), spent regenerator limestone (organics, trace elements, ESCA), gasifier bed stone (organics, trace elements, ESCA), left-hand cyclone particulate (organics and ESCA), right-hand cyclone particulates (trace elements and ESCA), knockout baffle particulates (organics, trace elements, and ESCA), and stack cyclone particulates (organics, trace elements, and ESCA). Methylene chloride was used to extract oil, particulates, and spent stone samples. Extraction procedures for other materials were not specified. The LC fractionation scheme specified in the Technical Manual for Analysis of Organic Materials in Process Streams, EPA, March 1976, was generally used in this study.

This LC procedure uses an eluent scheme different than that specified in Level 1 in that petroleum ether is used instead of pentane and also that 25-ml methanol is used to elute Fraction 8 rather than the specified 5/70/30 $HCl/MeOH/CH_2Cl_2$. Eluent volumes also differ. See LC schemes, Tables 3 and 4.

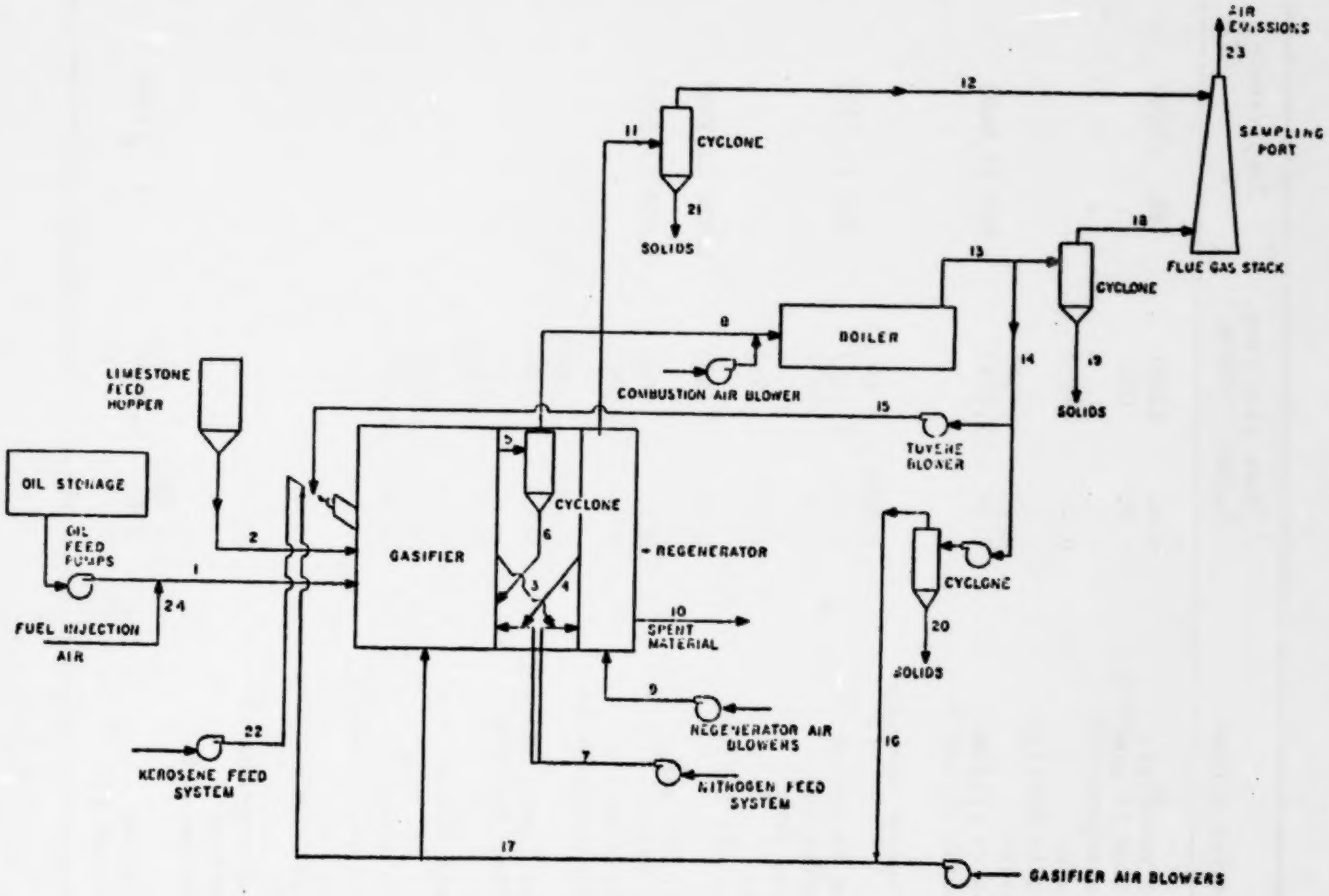
AIR EMISSIONS
23
12
SAMPLING PORT
11
CYCLONE
21
SOLIDS
18
FLUE GAS STACK
13
CYCLONE
8
BOILER
14
19
SOLIDS
LIMESTONE FEED HOPPER
COMBUSTION AIR BLOWER
15
TUYERE BLOWER
OIL STORAGE
5
CYCLONE
6
GASIFIER
REGENERATOR
2
OIL FEED PUMPS
1
3
4
CYCLONE
24
10
SPENT MATERIAL
FUEL INJECTION AIR
20
SOLIDS
9
REGENERATOR AIR BLOWERS
22
16
KEROSENE FEED SYSTEM
7
NITROGEN FEED SYSTEM
17
GASIFIER AIR BLOWERS

Figure 1. Unit operations flow diagram of the ERCA pilot plant.

TABLE 1. ERCA PILOT PLANT MASS FLOW RATES

Process stream	Mass flow rate, kg/sec	(lb/hr)	Temperature, °C	(°F)
1. Oil feed to gasifier	0.04	(288)	88	(190)
2. Limestone feed to gasifier	0.003	(25)		
3. Gasifier to regenerator stone transfer	0.11	(860)		
4. Regenerator to gasifier stone transfer	0.11	(850)		
5. Product gas to cyclone	0.16	(1,279)	850	(1,560)
6. Cyclone solids return to gasifier				
7. N_2 gas to solids transfer lines	0.0006	(4.5)		
8. Product gas to boiler	0.16	(1,279)	850	(1,560)
9. Air to regenerator	0.01	(65)		
10. Spent solids from regenerator	0.002	(14)		
11. Regenerator off gas to cyclone	0.01	(63)	1,050	(1,920)
12. Regenerator off gas, cyclone to stack	0.01	(63)	1,050	(1,920)
13. Flue gas from boiler	0.50	(4,000)		
14. Flue gas recirculated to gasifier	0.03	(250)		
15. Flue gas to Tuyere Blower	0.02	(125)		
16. Recycled flue gas from cyclone	0.02	(125)		
17. Flue gas and air to gasifier	0.10	(800)		
18. Flue gas to stack	0.50	(4,000)		
19. Solids from boiler flue gas cyclone				
20. Solids from recycled flue gas cyclone				
21. Solids from regenerator off gas cyclone				
22. Start up kerosene to gasifier	0.0005	(4)		
23. Stack emissions	0.50	(4,000)	43	(110)
24. Fuel injection air	0.01	(45)		

TABLE 2. SUMMARY OF SAMPLING ACTIVITY

	Run							
Type of sample or test	1	2	3	4	5	6	7	Other
SO_x	X	X			X			
NO_x	X	X			X			
H_2S	X	X	X					
Total particulate	X	X	X	X	X	X	X	
Particulate sizing			X		X			
O_2	X	X		X	X	X	X	
CO_2	X	X		X	X	X	X	
CO	X	X		X	X	X	X	
Moisture	X	X		X	X	X	X	
Organic stack gases			X	X		X	X	
Gasifier bed	X	X			X			
Regenerator bed		X	X	X	X			
Left-hand cyclone*		X	X	X				
Right-hand cyclone*					X			
Knockout baffle		X	X		X			
Stack cyclone			X	X	X			
Bitumen								X†
Fuel oil		X						
Limestone					X			X†
Leached stone								X

*These cyclones are located between the gasifier and boiler.
†Obtained during presampling site survey September 1975.

TABLE 3. LC SCHEME FROM TECHNICAL MANUAL FOR ANALYSIS OF ORGANIC MATERIALS IN PROCESS STREAMS

No. Fraction	Solvent composition	Volume of eluent (ml)
1	60/80 petroleum ether	25
2	20% methylene chloride in 60/80 petroleum ether	25
3	50% methylene chloride in 60/80 petroleum ether	25
4	Methylene chloride	25
5	5% methyl alcohol in methylene chloride	25
6	20% methyl alcohol in methylene chloride	25
7	50% methyl alcohol in methylene chloride	25
8	Methyl alcohol	25

TABLE 4. LC SCHEME FROM P. 128 OF THE LEVEL 1 ENVIRONMENTAL ASSESSMENTS PROCEDURES MANUAL (EPA-600/2-76-160a)

No. Fraction	Solvent composition	Volume of eluent (ml)
1	Pentane	25
2	20% methylene chloride in pentane	10
3	50% methylene chloride in pentane	10
4	Methylene chloride	10
5	5% methanol in methylene chloride	10
6	20% methanol in methylene chloride	10
7	50% methanol in methylene chloride	10
8	Conc. HCl/methanol/methylene chloride (5:7:30)	10

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY
STACK CYCLONE PARTICULATE
RUN 5, BITUMEN GASIFICATION (STARTUP)
(ppmw)

U		Dy	<0.2	Rh		Cr	31
Th	<0.2	Tb		Ru		V	1.04*
Ti	0.4	Gd		Mo	5.0	Ti	63
Pb	7.8	Eu		Nb	0.1	Sc	
Tl	0.2	Sm	<0.2	Zr	1.5	Ca	14.1%*
Hg		Nd	0.1	Y	0.2	K	340
Au		Pr	<0.1	Sr	32	Cl	120
Pt		Ce	0.6	Rb	1.1	S	3.83%†
Ir		La	0.2	Br	2.2	P	21
Os		Ba	55	Se	1.3	Si	0.49%
Re		Cs		As		Al	340
W	<0.3	I	1.3	Ge	4.1	Mg	0.32%
Ta	0.4	Te		Ga	0.4	Na	0.43%
Hf	<0.3	Sb		Zn	30	F	450
Lu		Sn	0.3	Cu	33	B	2.3
Yb	<0.5	In		Ni	0.22%*	Be	<0.12
Tm		Cd	0.3	Co	17	Li	4.3
Er		Ag		Fe	0.12%*		
Ho		Pd		Mn	21		

*Determined by atomic absorption spectrometry.
†Determined by wet chemistry.
No data indicates less than 0.1 ppmw.

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY
STACK CYCLONE PARTICULATE
RUN 4, FUEL OIL GASIFICATION
(ppmw)

U		Cy	0.2	Rh		Cr	31
Th	<0.2	Tb		Ru		V	0.80%*
Bi	<0.3	Gd		Mo	14	Ti	150
Pb	47	Eu		Nb	0.2	Sc	
Tl	<0.1	Sm	<0.2	Zr	3.0	Ca	34.5%*
Hg		Nd	0.2	Y	0.5	K	340
Au		Pr	0.1	Sr	180	Cl	0.16%
Pt		Ce	1.0	Rb	0.5	S	2.13%†
Ir		La	0.8	Br	60	P	96
Os		Ba	150	Se	1.3	Si	0.21%
Re		Cs		As		Al	0.13%
W	1.1	I	2.9	Ge	0.6	Mg	0.32%
Ta	0.4	Te		Ga	0.4	Na	850
Hf	<0.3	Sb		Zn	80	F	0.13%
Lu		Sn	1.0	Cu	44	B	5.0
Yb	<0.5	In		Ni	0.10%*	Be	<0.12
Tm		Cd	0.8	Co	5.0	Li	2.1
Er		Ag		Fe	0.15%*		
Ho		Pd		Mn	50		

*Determined by atomic absorption spectrometry.
†Determined by wet chemistry.
No data indicates less than 0.1 ppmw.

TABLE 7. GAS CHROMATOGRAPHY* FLUE GASES AFTER PARTICULATE REMOVAL

Sample	Flue gas flow rate (dscm/s)	Temp. at sampling port, °C	Moisture, %	CO_2 %	O_2 %	CO %	NO_x ppm	SO_2 ppm	SO_3 ppm	H_2S g/dscm	Total particulates g/dscm
Run 1 fuel oil gasification	0.56	108	8.3	13.0	1.0	0.1	53.5	292	8.3	.00007	0.117
Run 2 fuel oil gasification	0.51	109	8.6	12.0	3.8	0.1	45.7	305	7.9	.00007	0.073
Run 3 fuel oil gasification	0.60	111	9.7	12.2	2.8	0.2	---	---	---	.00032	0.080
Run 4 fuel oil gasification	0.58	133	9.0	11.6	5.3	0	---	---	---	---	0.106
Run 5 bitumen gasification (startup)	0.49	138	9.5	12.0	3.9	0	58.4	828	11.1	---	0.141
Run 6 bitumen gasification (w/lime feed)	0.56	80	2.4	12.0	3.9	0	---	---	---	---	0.056
Run 7 bitumen gasification	0.51	127	7.8	12.0	3.9	0	---	---	---	---	0.112

*Level 1 method of sampling and analysis was not used. Flue gases were sampled for NO_x by Federal Register Method 7, for SO_2/SO_3 by Method 8, for H_2S by Method 11, for CO, CO_2 and O_2 by Orsat.

TABLE 8. LC FRACTIONATION* VARIOUS PROCESS SAMPLES

	Bitumen feed				Flue gas after particle removal† run 7, bitumen gasification			
Fraction	TCO	Grav µg	Total	Total	TCO	Grav µg	Total	Total
LC 1		8,900				1,700		
LC 2		890				60		
LC 3		810				1,600		
LC 4		350				13,000		
LC 5		280				1,100		
LC 6		1,400				250		
LC 7		210				74		
LC 8		450				32		

Stack cyclone particulate
run 5, bitumen gasification (startup)‡

Fraction	TCO	Grav µg	Total	Total
LC 1		70		
LC 2		14		
LC 3		28		
LC 4		140		
LC 5		230		
LC 6		66		
LC 7		16		
LC 8		90		

See footnotes at end of table.

TABLE 8 (con.)

	Spent limestone‡ run 5, bitumen gasification (startup)				Stack cyclone particulates‡ run 4, fuel oil gasification			
Fraction	TCO	Grav µg	Total	Total	TCO	Grav µg	Total	Total
LC 1		330				97		
LC 2		64				210		
LC 3		82				28		
LC 4		110				120		
LC 5		85				180		
LC 6		290				200		
LC 7		57				35		
LC 8		210				0		

*By the LC fractionation scheme in Technical Manual for Analysis of Organic Materials in Process Streams.

†Flue gas was sampled for organics with a Tenax-GC cartridge, attached to a Method 5 train. A 1-hour sample was collected. Tenax-GC was extracted with pentane.

‡Extracted with methylene chloride.

TABLE 9. IR REPORT
SAMPLE: BITUMEN FEED*

LC	Wave number (cm^{-1})	Assignment (comments)
1	2,920; ~1,450; 1,370; ~1,600; 1,690	From CH_3, CH_2 Asphaltic materials (1690 band is from gross mixture of carbonyls and 1600 band is due to structures such as highly condensed aromatics and quinones.)
2	700-900	Same compounds as LC-1 but also contains: Aromatic compounds (possibly POM)
3		Same compounds as LC-1
4		Same compounds as LC-1
5	~3,400	Same compounds as LC-1 but also contains: -OH (bands at 1200-1300 suggest this might be phenol)
6	~1,025	Same compounds as LC-1 but also contains: probably SiO_2 impurity
7		Same as LC-6
8		Same as LC-6

*LC scheme from Technical Manual for Analysis of Organic Materials in Process Streams was used.

TABLE 10. IR REPORT
SAMPLE: FLUE GAS (AFTER PARTICLE REMOVAL)*
RUN 7, BITUMEN GASIFICATION

LC	Wave number (cm^{-1})	Assignments (comments)
1	2,920; 1,450; 1,370	Aliphatic hydrocarbons (Lower frequencies are silicone oil impurities.)
2		Same compounds as LC-1
3		Disubstituted amide, N-heteroaromatic, doubly conjugated ketone or quinone
4		Same compounds as LC-3
5		Same compounds as LC-3 plus:
	3,400	Alcohol or carboxylate
6	3,400	Carboxylate group
	1,640	Doubly conjugated ketone
	600-800	Aromatics
7		Same compounds as LC-1
8		Possible traces of carboxylic acid salts plus
	1,000-1,100	SiO_2 impurity

*Flue gas was sampled for organics with a Tenax-GC cartridge, attached to a Method 5 train. A 1-hour sample was collected. Tenax-GC was extracted with pentane. LC scheme from Technical Manual for Analysis of Organic Materials in Process Streams was used.

TABLE 11. IR REPORT
SAMPLE: STACK CYCLONE PARTICULATES*
RUN 4, FUEL OIL GASIFICATION

LC	Wave number (cm^{-1})	Assignments (comments)
1		Traces of aliphatic hydrocarbons
2	~2,920; 1,450; 1,370; 1,730; Peaks between 1,100-1,500	CH_3, CH_2 Carbonyl (C=0) group suggests aliphatic esters Other species containing C=0
3	~2,920; ~1,730	Aliphatic ester
4	~2,920; 1,450; 1,370 1,730 Broad band 1,000-1,100	Aliphatics } Probable aliphatic ester, ketone C=0 } or aldehyde Probably SiO_2 impurity
5	1,730	Carbonyl compounds
6	~2,920; 1,450; 1,370 ~3,400; 1,100-1,300	Aliphatic esters Carboxylates or alcohols
7	1,730 Broad band 1,000-1,100	Mixture of carbonyl compounds SiO_2 impurity

*Extracted with methylene chloride.

TABLE 12. IR REPORT
SAMPLE: STACK CYCLONE PARTICULATES*
RUN 5, BITUMEN GASIFICATION (STARTUP)

LC	Wave number (cm^{-1})	Assignment (comments)
1	~2,920; 1,450; 1,370	CH_3, CH_2 (aliphatic hydrocarbons)
	1,730	C=0
2	~2,920; 1,730	Aliphatic esters
3	1,730; ~1,500	Aliphatic carbonyl compounds
4	3,400	-OH
	600-800	Aromatics
	1,730	Carbonyls
	1,500, complex spectrum 1,000-1,300	Possible phthalates, phenols, or alcohols
5		Same as LC-4
6	---	Mixture of carbonyl and alcohol compounds
7&8		No data reported for these fractions

*Extracted with methylene chloride. LC scheme from Technical Manual for Analysis of Organics in Process Streams was used.

TABLE 13. IR REPORT
SAMPLE: REGENERATOR BED MATERIAL*
RUN 5, BITUMEN GASIFICATION (STARTUP)

LC	Wave number (cm^{-1})	Assignment (comments)
1	2,920; ~1,370; 1,450	CH_3, CH_2 (aliphatic hydrocarbons)
2		Same compounds as LC-1
3	 1,730	Same compounds as LC-1 plus: Ester (C=0)
4	 1,730 ~3,400	Same compounds as LC-1 plus: Carbonyl group Alcohol or carboxylate
5		Same compounds as LC-4
6	2,920	CH_3, CH_2
	1,730	C=0
	850, bands between 1,000-1,600	Aromatic carbonyl compounds
	3,400	Phenol or carboxylic acid
7	----	Traces of aliphatics and carbonyl compounds
8		Same compounds as LC-6

*Extracted with methylene chloride. LC scheme from Technical Manual for Analysis of Organics in Process Streams was used.

STUDY NUMBER 2

DATA SOURCE:

FLUE GAS SAMPLING DURING THE COMBUSTION OF SOLVENT REFINED COAL IN A UTILITY BOILER

DATA STATUS: Preliminary Report, July 1977

AUTHORS: C. S. Koralek and V. Bruce May

CONTRACTOR:
HITTMAN ASSOCIATES, INC.
9190 Red Branch Road
Columbia, Maryland 21045
Contract No. 68-02-2162

PROJECT OFFICER:
Bill Rhodes
Industrial Environmental Research Laboratory
Fuel Process Branch
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

This study at Georgia Power Company's Plant Mitchell in Albany, Georgia, was designed to determine if solvent-refined coal (SRC) could replace coal as a primary fuel in a pulverized coal-fired boiler. Hittman Associates, Inc., acted as coordinator of part of the test programs. The study was conducted in three phases utilizing: I--low-sulfur coal; II--modified burners and low-sulfur coal; and III--modified burners and SRC. A preliminary report evaluating available results indicates the following:

1. SRC can be a suitable replacement fuel for conventional coal in pulverized coal-fired boilers;
2. Averaging results of two analytical techniques for SO_x indicate SO_x emissions approximately equal when burning SRC or regular coal. Overall emissions are greatest for SRC at maximum load;
3. NO_x levels for SRC are about 10 percent greater than NO_x levels using conventional pulverized coal, with NO_x emission levels being greatest at maximum load;
4. C_1-C_6 organic emissions from SRC burners are negligible; and
5. SRC particulates in this study are higher in carbon than conventional coal boiler particulates (70 percent v. 10 percent), and the SRC flyash is comparatively smaller (20 percent of SRC flyash <1 μm compared to 2-5 percent of conventional coal flyash <1 μm). The SRC flyash has a high resistivity and correspondingly low electrostatic precipitator collection efficiency indicating possible future control technology problems.

GASEOUS GRAB

Tedlar bags were used for gaseous grab samples rather than glass bulbs. In phases II and III of the study, gaseous grab samples were analyzed for C_1-C_6 organics by Level I methods (GC-FID) and for O_2, N_2, CO, CO_2, and SO_x by GC with thermal conductivity detection as specified in Level I procedures. NO_x and SO_x were also continuously monitored using a Thermal Electron NO_x analyzer and a Theta Sensor SO_x monitor.

SASS

A SASS train was used to sample flue gases in phases II and III. Results of analysis on SASS samples were not yet available at the time this report was published.

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FUGITIVE EMISSIONS

Not sampled in this study.

LIQUIDS AND SLURRIES

Not sampled in this study.

SOLIDS

Daily composites were prepared of coal and SRC from phases II and III. Bottom ash samples from the combustion chamber were also collected. Analytical results are not yet available.

TABLE 1. GAS CHROMATOGRAPHY FOR INORGANIC GASES
PHASE II--LOW SULFUR COAL

Date	Sample Load conditions*	Sample Sampling site†	SO_2‡ (ppm)	CO§ (ppm)	O_2# (%)	CO_2# (%)	N_2# (%)
5/26	Low	0-1	254/260	ND	13.31	7.40	79.29
5/31	Low	0-1	329/360	ND	14.24	7.50	78.26
6/02	Low	I-1	174/200	ND	14.91	6.56	78.53
5/25	Med	0-1	413/500	ND	15.73	5.51	78.76
5/29	Med	I-1	209/220	ND	13.70	7.59	78.71
5/30	Med	0-1	413/400	ND	12.60	7.35	80.05
5/27	Full	0-1	311/330	ND	13.78	6.65	79.66
5/28	Full	I-1	381/330	ND	11.25	9.86	78.89
6/05	Full	0-3	214/200	ND	12.14	9.31	78.55
6/06	Full	0-3	210/180	ND	11.16	9.69	79.15

*Low output = 7 MW
Med output = 14 MW
Full output = 21 MW.

†Sampling sites: 0-1 = outlet to precipitator 1
I-1 = inlet to precipitator 1
0-3 = outlet to precipitator 3.

‡First number indicates onsite GC (±2%); second number indicates continuous monitor (Theta Sensor) (±10 ppm).

§40 ppm minimum detection limit; ND = none detected.

±2% of total concentration.

TABLE 2. GAS CHROMATOGRAPHY FOR INORGANIC GASES PHASE III--SOLVENT REFINED COAL

Date	Sample Load conditions*	Sampling site†	SO_2‡ (ppm)	CO§ (ppm)	O_2# (%)	CO_2# (%)	N_2# (%)
6/15	Low	0-1	198/225	ND	14.79	5.88	79.33
6/18	Low	I-1	216/220	ND	13.25	6.73	80.02
6/19	Low	0-1	218/235	ND	14.00	6.26	79.74
6/14	Med	0-1	248/260	ND	13.65	7.53	78.82
6/13	Full	0-1	371/325	ND	11.39	9.86	78.75
6/16	Full	0-1	410/335	ND	10.62	9.12	80.26
6/17	Full	I-1	404/345	ND	11.11	9.15	79.74
6/22	Full	0-3	400/345	ND	11.20	9.25	79.55
6/23	Full	0-3	393/325	ND	10.75	8.90	80.35
6/24	23.5 MW	0-1	449/380	ND	10.76	9.29	79.95

*Low output = 7 MW
Med output = 14 MW
Full output = 21 MW.

†Sampling sites: 0-1 = outlet to precipitator 1
I-1 = inlet to precipitator 1
0-3 = outlet to precipitator 3.

‡First number indicates onsite GC (±2%); second number indicates continuous monitor (Theta Sensor) (±10 ppm).

§40 ppm minimum detection limit; ND = none detected.

# ±2% of total concentration.

TABLE 3. CHEMILUMINESCENCE FOR NO_x DURING PHASE III--SOLVENT REFINED COAL

Sample			NO_x concentrations
Date	Load conditions*	Sampling site†	(ppm)‡
6/15	Low	0-1	125
6/18	Low	I-1	120
6/19	Low	0-1	125
6/14	Med	0-1	160
6/13	Full	0-1	190
6/16	Full	0-1	190
6/17	Full	I-1	190
6/22	Full	0-3	200
6/23	Full	0-3	220
6/24	23.5 MW	0-1	260

*Low output = 7 MW
Med output = 14 MW
Full output = 21 MW.

†Sampling sites: 0-1 = outlet to precipitator 1
I-1 = inlet to precipitator 1
0-3 = outlet to precipitator 3.

‡ ±10 ppm.

TABLE 4. CHEMILUMINESCENCE FOR NO_x DURING PHASE II--LOW SULFUR COAL

Sample			NO_x concentrations
Date	Load conditions*	Sampling site†	(ppm)‡
5/26	Low	0-1	110
5/31	Low	0-1	110
6/02	Low	I-1	100
5/25	Med	0-1	170
5/29	Med	I-1	160
5/30	Med	0-1	150
5/24	Full	0-1	225
5/27	Full	0-1	215
5/28	Full	I-1	220
6/05	Full	0-3	170
6/06	Full	0-3	110

*Low output = 7 MW
Med output = 14 MW
Full output = 21 MW.

†Sampling sites: 0-1 = outlet to precipitator 1
I-1 = inlet to precipitator 1
0-3 = outlet to precipitator 3.

‡ ±10 ppm.

TABLE 5. GAS CHROMATOGRAPHY FOR C_1-C_6/C_7
PHASE II--LOW SULFUR COAL

Sample	Range	Weight	Number of peaks observed
5/25 Medium output (14 MW) Outlet to precipitator 1	GC1 -160 - -100	NONE DETECTED*	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
5/29 Medium output (14 MW) Inlet to precipitator 1	GC1 -160 - -100	NONE DETECTED†	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
5/30 Medium output (14 MW) Outlet to precipitator 1	GC1 -160 - -100	NONE DETECTED†	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
5/26 Low output (7 MW) Outlet to precipitator 1	GC1 -160 - -100	NONE DETECTED*	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
5/31 Low output (7 MW) Outlet to precipitator 1	GC1 -160 - -100	NONE DETECTED†	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
6/02 Low output (7 MW) Inlet to precipitator 1	GC1 -160 - -100	NONE DETECTED†	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		

See footnotes at end of table.

TABLE 5 (con.)

Sample	Range	Weight	Number of peaks observed
5/27 Full output (21 MW) Outlet to precipitator 1	GC1 -160 - -100	NONE DETECTED*	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
5/28 Full output (21 MW) Inlet to precipitator 1	GC1 -160 - -100	NONE DETECTED†	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
6/05 Full output (21 MW) Outlet to precipitator 3	GC1 -160 - -100	NONE DETECTED†	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
6/06 Full output (21 MW) Outlet to precipitator 3	GC1 -160 - -100	NONE DETECTED†	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		

*5 ppm = minimum detectable limit.
†0.5 ppm = minimum detectable limit.

TABLE 6. GAS CHROMATOGRAPHY FOR C_1-C_6/C_7 PHASE III--SOLVENT REFINED COAL

Sample	Range	Weight*	Number of peaks observed
6/15	GC1 -160 - -100	NONE DETECTED	
Low output	GC2 -100 - -50		
(7 MW)	GC3 -50 - 0		
Outlet to	GC4 0 - 30		
precipitator 1	GC5 30 - 60		
	GC6 60 - 90		
6/18	GC1 -160 - -100	NONE DETECTED	
Low output	GC2 -100 - -50		
(7 MW)	GC3 -50 - 0		
Inlet to	GC4 0 - 30		
precipitator 1	GC5 30 - 60		
	GC6 60 - 90		
6/19	GC1 -160 - -100	NONE DETECTED	
Low output	GC2 -100 - -50		
(7 MW)	GC3 -50 - 0		
Outlet to	GC4 0 - 30		
precipitator 1	GC5 30 - 60		
	GC6 60 - 90		
6/14	GC1 -160 - -100	NONE DETECTED	
Medium output	GC2 -100 - -50		
(14 MW)	GC3 -50 - 0		
Outlet to	GC4 0 - 30		
precipitator 1	GC5 30 - 60		
	GC6 60 - 90		
6/13	GC1 -160 - -100	NONE DETECTED	
Full output	GC2 -100 - -50		
(21 MW)	GC3 -50 - 0		
Outlet to	GC4 0 - 30		
precipitator 1	GC5 30 - 60		
	GC6 60 - 90		
6/16	GC1 -160 - -100	NONE DETECTED	
Full output	GC2 -100 - -50		
(21 MW)	GC3 -50 - 0		
Outlet to	GC4 0 - 30		
precipitator 1	GC5 30 - 60		
	GC6 60 - 90		

See footnote at end of table.

TABLE 6 (con.)

Sample	Range	Weight*	Number of peaks observed
6/17 Full output (21 MW) Inlet to precipitator 1	GC1 -160 - -100	NONE DETECTED	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
6/22 Full output (21 MW) Outlet to precipitator 3	GC1 -160 - -100	NONE DETECTED	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
6/23 Full output (21 MW) Outlet to precipitator 3	GC1 -160 - -100	NONE DETECTED	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		
6/24 Output = 23.5 MW Outlet to precipitator 1	GC1 -160 - -100	NONE DETECTED	
	GC2 -100 - -50		
	GC3 -50 - 0		
	GC4 0 - 30		
	GC5 30 - 60		
	GC6 60 - 90		

*0.5 ppm = minimum detectable limit.

STUDY NUMBER 3

DATA SOURCE:

SASS TRAIN AND LEVEL I PROCEDURES EVALUATION

DATA STATUS: Final Report, November 1976

AUTHORS: S. L. Reynolds
D. G. Ackerman, J. F. Clausen, M. L. Kraft, C. A. Zee

CONTRACTOR: TRW Defense and Space Systems Group
One Space Park
Redondo Beach, California 90278
Contract No. 68-02-2165, Task 14

PROJECT OFFICER: Dr. R. M. Statnik
Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

GENERAL

The purpose of this study, performed at the coal-fired boiler at the KVB R&D facility in Tustin, California, was to evaluate the trapping efficiency of the SASS train. The raw materials, control device systems, and all parts of the SASS train were analyzed to perform mass balance determinations.

The actual catch from the SASS train was;

Probe solids	0.0939 g
10μ cyclone	38.5477
3μ cyclone	23.7844
1μ cyclone	11.2979
Filters (3)	3.3463
	Σ=77.0702

Sampling was at 4 actual cfm rather than 4 standard cfm.

Several suggestions were made for improvements on the SASS train design. There were problems with galling or cross-threading of the interconnect fittings within the cyclone system. Gyrolok fittings, AN fittings, stainless steel ball and socket joints, or welding of some of the interconnect tubes were suggested to solve these problems. Difficulties with the clamping of the upper and lower halves of the 10μ cyclone and with cleaning of the 3μ cyclone reservoir were discussed. The exit tube to the 1μ cyclone tended to shear off when it was used as a lever to tighten the fittings; two possible solutions to this problem involve reinforcement of the joint weld or louvering the outer surface of the cup and upper portion of the cyclone to provide a firmer grip. Because of the necessity of frequent filter changes when flat filters are used, "S"-pleated filters were suggested. It was further suggested that lowering the XAD-2 module temperature from 60° C to 30° C would reduce corrosion problems. In the impingers system, Lexan was recommended as a preferable construction material and it was also recommended that the pressure equalization tubes be closed off to permit a smaller impinger solution volume. Use of an insulated impinger container and a higher-capacity desiccant were proposed.

Elemental analyses in this study were performed using SSMS, AAS, and electron microprobe (EM). Cl^- and F^- analyses were by specific ion electrode. Four methods of preparation of samples for inorganic analysis were investigated: Parr bomb combustion, Teflon bomb digestion with HNO_3:HF, Teflon bomb digestion will HNO_3, and Teflon bomb digestion with HNO_3:H_2O_2. It was concluded that Parr bomb combustion "released more metal than acid leaching" and was the method of choice.

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SSMS analyses were used in mass balance determination and in characterizing elemental capture by the various components of the SASS train. Elements were generally distributed as was expected, the heavy metals concentrating toward the 10μ cyclone and the volatile elements concentrating toward the impingers. An unanticipated effect was the trapping of volatile trace elements by the condenser well. Mass balance closure was stated to be generally good with the exception of contaminants from the 316 stainless steel parts of the SASS train.

Specific elemental analysis was performed for As, Hg, Sb, Se, Cd, Pb, and V. As analysis was by the SDDC (colorimetric) method, and Hg analysis was by flameless AA. The remainder of the elements were analyzed by standard AA techniques.

Organic analyses were by Level 1 procedures with a few modifications. GC analysis for C_7-C_{12} volatile organics was performed with room temperature injection programmed from 29° C to 150° C at 10° C/min rather than the specified 5 minutes at 50° C with programming from 50° C to 150° C at 10° C/min. After LC fractionation, a colored material remained in the column and three more fractions were collected (10 ml of $HCl/CH_3OH/CH_2Cl_2$, 10 ml of CH_3OH and 10 ml of acetonitrile) to determine the nature of the colored material. On IR analysis of three samples, fraction 9 showed water, alcohol, aromatics, and an aromatic ketone. Fraction 10 showed water, alcohol, sulfonic acid, and an aromatic ketone. Fraction 11 showed nothing by IR analysis.

Particle characterization was performed on two filters. Photomicrographs and accompanying comments from observation at 42X magnification were presented.

GASEOUS GRAB

No sampling was performed in this study.

SASS

Cyclone and filter catches were analyzed individually by SSMS, organic techniques, and specific ion electrode to determine distribution of constituents through the sampling system. The Level 1 specified CH_2Cl_2:CH_3OH rinse was followed by an acetone rinse, then a benzene rinse, to test the efficiency of the Level 1 solvent rinses. The XAD-2 cannister rinse was analyzed separately from the condenser well to differentiate between organics trapped in these two stages. The condenser well was also rinsed with 1:1 HNO_3:H_2O to determine what trace metals were trapped there.

The impinger system was evaluated for several parameters. Trapping efficiency of the H_2O_2-APS solution at different temperatures was determined to be 100 percent in a study involving an Hg stream. A concurrent stability study showed that after 12 hours, trapping efficiency was 0 percent. A black precipitate was noted in the second and third impingers after the sample had been collected. Microprobe analysis showed that this precipitate was silver sulfide.

FUGITIVE EMISSIONS

No sampling was performed in this study.

LIQUIDS AND SLURRIES

No sampling was performed in this study.

SOLIDS

Coal feed, bottom ash, and baghouse flyash were sampled and analyzed as indicated in Figure 1 taken from the study:

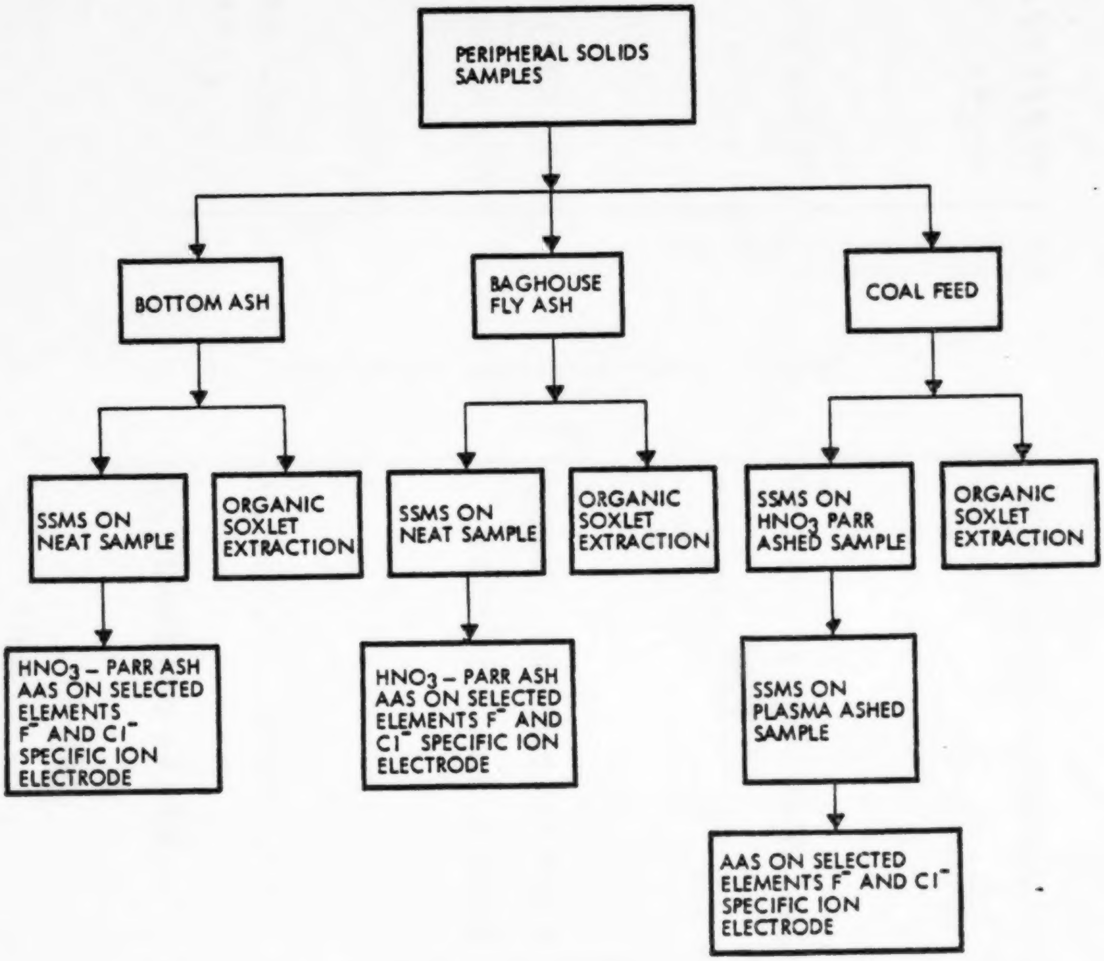
PERIPHERAL SOLIDS SAMPLES
BOTTOM ASH
BAGHOUSE FLY ASH
COAL FEED
SSMS ON NEAT SAMPLE
ORGANIC SOXLET EXTRACTION
SSMS ON NEAT SAMPLE
ORGANIC SOXLET EXTRACTION
SSMS ON HNO3 PARR ASHED SAMPLE
ORGANIC SOXLET EXTRACTION
HNO3 – PARR ASH AAS ON SELECTED ELEMENTS F⁻ AND Cl⁻ SPECIFIC ION ELECTRODE
HNO3 – PARR ASH AAS ON SELECTED ELEMENTS F⁻ AND Cl⁻ SPECIFIC ION ELECTRODE
SSMS ON PLASMA ASHED SAMPLE
AAS ON SELECTED ELEMENTS F⁻ AND Cl⁻ SPECIFIC ION ELECTRODE

Figure 1. Analysis plan for peripheral solids samples.

TABLE 1. SPARK SOURCE MASS SPECTROSCOPY
PLASMA-ASHED COAL FEED
(μg/g)

U	9	Dy	6	Rh		Cr	340
Th	15	Tb	1	Ru		V	420
Bi	≤0.8	Gd	5	Mo	10	Ti	MC
Pb	36	Eu	2	Nb	23	Sc	75
Tl	6	Sm	5	Zr	230	Ca	MC
Hg		Nd	20	Y	2	K	MC
Au		Pr	5	Sr	MC	Cl	50
Pt		Ce	110	Rb	97	S	MC
Ir		La	37	Br	2	P	MC
Os		Ba	320	Se	6	Si	MC
Re		Cs	5	As	20	Al	MC
W	≤1	I	2	Ge	3	Mg	MC
Ta		Te		Ga	33	Na	MC
Hf		Sb	10	Zn	79	F	MC
Lu	0.3	Sn	4	Cu	60	B	940
Yb	89	In		Ni	45	Be	7
Tm	0.4	Cd	2	Co	20	Li	190
Er	3	Ag	≤4	Fe	MC		
Ho	2	Pd		Mn	240		

No data indicates not reported.
MC = major component.

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY
BOTTOM ASH
(μg/g)

U	4	Dy	2	Rh		Cr	340
Th	11	Tb	0.7	Ru		V	110
Bi	0.6	Gd	1	Mo	9	Ti	MC
Pb	16	Eu	0.5	Nb	10	Sc	42
Tl	1	Sm	2	Zr	110	Ca	MC
Hg		Nd	9	Y	40	K	MC
Au		Pr	5	Sr	960	Cl	280
Pt		Ce	52	Rb	87	S	MC
Ir		La	20	Br	8	P	MC
Os		Ba	290	Se	2	Si	MC
Re		Cs	5	As	17	Al	MC
W	0	I	9	Ge	3	Mg	MC
Ta		Te		Ga	13	Na	MC
Hf		Sb	0.9	Zn	70	F	~1,180
Lu	0.1	Sn	2	Cu	75	B	130
Yb	0.9	In		Ni	140	Be	4
Tm	0.2	Cd	0.7	Co	30	Li	*
Er	0.6	Ag	0.3	Fe	MC		
Ho	1	Pd		Mn	320		

No data indicates not reported.
MC = major component.
*Interference

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
1μ-3μ PARTICULATES
(μg/g)

U	4	Dy	2	Rh		Cr	290
Th	≤0.4	Tb	0.7	Ru		V	200
Bi	0.7	Gd	1	Mo	8	Ti	MC
Pb	39	Eu	0.6	Nb	11	Sc	45
Tl	2	Sm	2	Zr	280	Ca	MC
Hg		Nd	5	Y	42	K	MC
Au		Pr	3	Sr	MC	Cl	300
Pt		Ce	100	Rb	100	S	MC
Ir		La	†	Br	1	P	MC
Os		Ba	300	Se	0.7	Si	MC
Re		Cs	†	As	100	Al	MC
W	1	I	17	Ge	12	Mg	MC
Ta		Te		Ga	70	Na	MC
Hf	1	Sb	3	Zn	190	F	~75
Lu	0.2	Sn	5	Cu	36	B	MC
Yb	1	In		Ni	35	Be	8
Tm	0.3	Cd	1	Co	32	Li	22
Er	0.9	Ag	0.3	Fe	MC		
Ho	1	Pd		Mn	50		

No data indicates not reported.
†Value less than blank value.
MC = major component.

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
3μ-10μ PARTICULATES
(μg/g)

U	4	Dy	3	Rh		Cr	450
Th	11	Tb	1	Ru		V	180
Bi	0.7	Gd	2	Mo	9	Ti	MC
Pb	13	Eu	0.8	Nb	17	Sc	68
Tl	†	Sm	2	Zr	170	Ca	MC
Hg		Nd	9	Y	80	K	MC
Au		Pr	4	Sr	780	Cl	MC
Pt		Ce	130	Rb	70	S	MC
Ir		La	34	Br	2	P	MC
Os		Ba	290	Se	1	Si	MC
Re		Cs	5	As	68	Al	MC
W	≤0.8	I	8	Ge	7	Mg	MC
Ta		Te		Ga	43	Na	MC
Hf	≤0.9	Sb	2	Zn	57	F	~76
Lu	0.3	Sn	6	Cu	44	B	680
Yb	3	In		Ni	52	Be	7
Tm	0.3	Cd	2	Co	24	Li	3
Er	1	Ag	1	Fe	MC		
Ho	1	Pd		Mn	92		

No data indicates not reported.
†Value less than blank value.
MC = major component.

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY
>10μ PARTICULATES
(μg/g)

U	4	Dy	2	Rh		Cr	490
Th	20	Tb	1	Ru		V	200
Bi	1	Gd	2	Mo	9	Ti	MC
Pb	26	Eu	0.8	Nb	11	Sc	60
Tl	1	Sm	2	Zr	180	Ca	MC
Hg		Nd	10	Y	47	K	MC
Au		Pr	4	Sr	MC	Cl	MC
Pt		Ce	56	Rb	92	S	MC
Ir		La	55	Br	1	P	MC
Os		Ba	510	Se	2	Si	MC
Re		Cs	4	As	45	Al	MC
W	0.6	I	3	Ge	5	Mg	MC
Ta		Te		Ga	31	Na	MC
Hf	2	Sb	0.8	Zn	38	F	~190
Lu	0.2	Sn	2	Cu	48	B	MC
Yb	2	In		Ni	58	Be	7
Tm	0.3	Cd	2	Co	18	Li	≥180
Er	0.9	Ag	0.2	Fe	MC		
Ho	1	Pd		Mn	340		

No data indicates not reported.
MC = major component.

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY
BAGHOUSE ASH
(μg/g)

U	4	Dy	2	Rh		Cr	160
Th	18	Tb	0.6	Ru		V	180
Bi	0.4	Gd	1	Mo	8	Ti	MC
Pb	35	Eu	43	Nb	20	Sc	40
Tl	1	Sm	2	Zr	200	Ca	MC
Hg		Nd	20	Y	75	K	MC
Au		Pr	8	Sr	MC	Cl	530
Pt		Ce	89	Rb	82	S	MC
Ir		La	39	Br	7	P	MC
Os		Ba	270	Se	1	Si	MC
Re		Cs	19	As	40	Al	MC
W	0.09	I	18	Ge	9	Mg	MC
Ta		Te		Ga	25	Na	MC
Hf	0.9	Sb	2	Zn	2.0	F	~170
Lu	0.2	Sn	4	Cu	43	B	710
Yb	1	In		Ni	78	Be	3
Tm	0.1	Cd	0.9	Co	23	Li	53
Er	0.6	Ag	4	Fe	MC		
Ho	1	Pd		Mn	≥70		

No data indicates not reported.
MC = major component.

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY
FILTER NUMBER 2
(μg/g)

U	29	Dy	4	Rh		Cr	394
Th	10	Tb	3	Ru		V	551
Bi	2	Gd	12	Mo	43	Ti	≥687
Pb	55	Eu	6	Nb	50.2	Sc	153
Tl	6	Sm	10	Zr	90	Ca	†
Hg		Nd	26	Y	29	K	†
Au		Pr	11.3	Sr	≥898	Cl	0
Pt		Ce	214	Rb	55.1	S	≥796
Ir		La	66	Br	3.4	P	≥801
Os		Ba	493	Se	≤0.31	Si	†
Re		Cs	8.8	As	41.7	Al	†
W	2	I	0	Ge	10	Mg	†
Ta		Te		Ga	104.3	Na	†
Hf		Sb	6	Zn	116	F	MC
Lu		Sn	18	Cu	22.2	B	†
Yb		In		Ni	0	Be	36.9
Tm		Cd	0	Co	410	Li	261.3
Er		Ag	0	Fe	MC		
Ho	2	Pd		Mn	140.2		

No data indicates not reported.
MC = major component.
†Value less than blank value.

TABLE 8. SPARK SOURCE MASS SPECTROSCOPY
FILTER NUMBER 3
(μg/g)

U	0.2	Dy	0.05	Rh		Cr	†
Th	0.2	Tb	0.2	Ru		V	5
Bi	0.02	Gd	.08	Mo	†	Ti	≥687
Pb	†	Eu	0.02	Nb	†	Sc	†
Tl	0.06	Sm	0.1	Zr	†	Ca	†
Hg		Nd	†	Y	†	K	†
Au		Pr	†	Sr	≥898	Cl	†
Pt		Ce	†	Rb	†	S	≥796
Ir		La	†	Br	†	P	≥801
Os		Ba	≥703	Se	≥0.5	Si	†
Re		Cs	†	As	†	Al	†
W	†	I	†	Ge	10	Mg	†
Ta		Te		Ga	†	Na	†
Hf	†	Sb	0.1	Zn	†	F	
Lu	†	Sn	0.1	Cu	†	B	†
Yb	†	In		Ni	†	Be	0.1
Tm	0.006	Cd	†	Co	4	Li	
Er		Ag	†	Fe			
Ho	.02	Pd		Mn	†		

No data indicates not reported.
MC = major component.
†Value less than blank value.

TABLE 9. SPARK SOURCE MASS SPECTROSCOPY
XAD-2 EXTRACT
(μg/ml)

U		Dy		Rh		Cr	3
Th		Tb		Ru		V	†
Bi		Gd		Mo	0	Ti	0
Pb	5	Eu	0.003	Nb	1	Sc	0
Tl	0.08	Sm	0.005	Zr	†	Ca	1
Hg		Nd	.006	Y		K	†
Au		Pr	.003	Sr	0.1	Cl	†
Pt		Ce	.013	Rb	†	S	†
Ir		La	0.001	Br	$\leq$0.002	P	0.2
Os		Ba	0.16	Se	0	Si	†
Re		Cs	*0	As	†	Al	0.1
W		I		Ge		Mg	0
Ta		Te		Ga	.002	Na	†
Hf		Sb		Zn	0.1	F	†
Lu		Sn	0.013	Cu	6.5	B	†
Yb		In		Ni	$\geq$2	Be	
Tm		Cd		Co	†	Li	†
Er		Ag	.01	Fe	3		
Ho		Pd		Mn	0		

*Corrected for blank.
†Value less than blank value.
No data indicates not reported.

TABLE 10. SPARK SOURCE MASS SPECTROSCOPY
XAD-2 MODULE HNO_3 RINSE
(μg/ml)

U		Dy		Rh		Cr	MC
Th		Tb		Ru		V	1
Bi		Gd		Mo		Ti	1
Pb	$\leq$0.09	Eu		Nb		Sc	0.09
Tl		Sm		Zr		Ca	6
Hg		Nd		Y		K	1
Au		Pr		Sr	0.02	Cl	0.7
Pt		Ce		Rb	0.01	S	MC
Ir		La		Br	.02	P	8
Os		Ba	0.03	Se	0.07	Si	MC
Re		Cs	*0.01	As	.6	Al	0.4
W		I		Ge	.02	Mg	MC
Ta		Te		Ga	.06	Na	MC
Hf		Sb	0.03	Zn	0.2	F	~2
Lu		Sn		Cu	2	B	0.9
Yb		In		Ni	MC	Be	
Tm		Cd	0.06	Co	2	Li	.002
Er		Ag	.04	Fe	MC		
Ho		Pd		Mn	2		

No data indicates not reported.
MC = major component.

TABLE 11. SPARK SOURCE MASS SPECTROSCOPY IMPINGERS (μg/ml)

U		Dy		Rh		Cr	2
Th		Tb		Ru		V	≤0.02
Bi		Gd		Mo	0.08	Ti	≤0.2
Pb	≤0.05	Eu		Nb		Sc	≤0.01
Tl		Sm		Zr	≤0.02	Ca	1.2
Hg		Nd		Y	†	K	‡
Au		Pr		Sr	†	Cl	0.2
Pt		Ce	0.01	Rb	0.02	S	MC
Ir		La	0.003	Br	≤0.003	P	2
Os		Ba	0.002	Se	≤0.005	Si	1.6
Re		Cs	2	As	≤0.04	Al	0.05
W		I		Ge		Mg	0.6
Ta		Te		Ga	.05	Na	MC
Hf		Sb		Zn	0.4	F	†
Lu		Sn		Cu	0.08	B	0
Yb		In		Ni	0.5	Be	
Tm		Cd		Co	.01	Li	*
Er		Ag		Fe	5		
Ho		Pd		Mn	0.1		

*Interference.

†Value less than blank value.

‡Blank and sample both contained potassium as a major component.

No data indicates not reported.

MC = major component.

TABLE 12. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS*
(μg/g)

Sample	Hg	Sb	As
Benzoic acid blank	---	<30	<3
Bottom ash (composite)	0.04	<60	<6
10μ cyclone	0.20	<290	<29
3μ cyclone	0.24	<290	29
1μ cyclone	1.39	<300	50
XAD-2 blank	0.10	<15	<1.5
XAD-2 sample	0.18	<15	1.5
Baghouse ash	0.18	<290	<29
Coal feed	0.007	<30	<3
HNO_3 wash of XAD-2 module	---	1.4 μg/ml	8 μg/ml
Impinger #1	0.00085 μg/ml	3 μg/ml	0.06 μg/ml
Impinger #2	0.0071 μg/ml	<0.2 μg/ml	<0.06 μg/ml
Impinger #3	0.0062 μg/ml	<0.2 μg/ml	<0.06 μg/ml

*Analysis by AAS.

TABLE 13. ANION ANALYSIS*

Sample	Cl μg/g	F μg/g	SO_4	SO_3	NO_3	NO_2
Coal feed	2	<1				
Baghouse ash	2	<1				
Bottom ash	0.8	<1				
10μ cyclone	2	<1				
3μ cyclone	8	<1				
1μ cyclone	27	<1				
XAD-2 sample	15	<1				
XAD-2 blank	5	<1				
Impinger blank	0	<1				
Impinger #1	1,362†	<1				

*By specific ion electrode.

†This value is mg in total impinger.

TABLE 14. LC FRACTIONATION

Fraction	XAD-2 module wash* TCO	Grav mg	Total	Total	XAD-2 cannister wash† TCO	Grav mg	Total	Total
LC 1		0.0				0.0		
LC 2		0.0				0.0		
LC 3		0.0				0.0		
LC 4		0.0				0.0		
LC 5		0.0				0.0		
LC 6		0.197				0.356		
LC 7		13.544				32.868		
LC 8		51.438				30.679		

Fraction	Coal feed extract‡ TCO	Grav mg	Total	Total
LC 1		3.798		
LC 2		1.576		
LC 3		9.906		
LC 4		3.190		
LC 5		1.884		
LC 6		8.543		
LC 7		3.070		
LC 8		11.438		

---

*Used 10 ml of sample, containing 161.5 mg of gravimetric solids.
†Used 20 ml of sample, containing 196.6 mg of gravimetric solids.
‡Used 50 ml of sample, containing 50.5 mg of gravimetric solids.

TABLE 15. IR REPORT
SAMPLE: XAD-2 MODULE WASH

LC Fraction	Assignments
1	No IR
2	No IR
3	No IR
4	No IR
5	No IR
6	No IR
7	Water/alcohol, possibly aromatic ketone
8	Water/alcohol; silicone, aromatic ketone

TABLE 16. IR REPORT
SAMPLE: XAD-2 CANNISTER WASH

LC Fraction	Assignments
1	No IR
2	No IR
3	No IR
4	No IR
5	No IR
6	No IR
7	Water/alcohol
8	Water/alcohol

TABLE 17. IR REPORT
SAMPLE: COAL FEED EXTRACT

LC Fraction	Assignments
1	Alkanes, trace silicone
2	Alkanes, alkenes, trace silicone, trace aromatics
3	Ethers
4	Water, olefins, ethers, silicones, aromatics
5	Water, aromatics, ethers
6	Water, ethers
7	Water, ethers
8	Water, sulfonic acid

TABLE 18. LRMS REPORT
PENTANE BLANK PLUS CONDENSATE BLANK BEFORE LC FRACTIONATION

I. Categories Present

Intensity	Category	MW range
Major	Phthalate esters	---
Minor	$C_7H_{13}O_2$ (esters, acids, etc.) or $C_{10}H_9$ (indanes, indenes, or tetrahydronaphthalenes)	---

TABLE 19. LRMS REPORT
COMBINED CYCLONE RINSES BEFORE LC FRACTIONATION

I. Categories Present

Intensity	Category	MW range
Major	Phthalate esters	---
Minor	Aliphatic hydrocarbons	200

TABLE 20. LRMS REPORT
XAD-2 RESIN EXTRACT BEFORE LC FRACTIONATION

I. Categories Present

Intensity	Category	MW range
Major	Phthalate esters believed to be primarily dioctylphthalate	---
Moderate	Ethyl benzoate	---

TABLE 21. LRMS REPORT
BAGHOUSE DUST BEFORE LC FRACTIONATION

I. Categories Present

Intensity	Category	MW range
Major	Hydrocarbons, including alkanes, alkenes, dienes, cyclics	---
Moderate	C_3H_7-O-fragment (ether, alcohol, ester)	---
Trace	Possible biphenyl or acenaphthalene	---
Trace	Possible naphthoic acid	---
Trace	Phthalate esters	

TABLE 22. LRMS REPORT
BOTTOM ASH BEFORE LC FRACTIONATION

I. Categories Present

Intensity	Category	MW range
Major	Phthalate esters	---
Major	Hydrocarbons including alkanes, alkenes, and possible dienes or cycloalkenes	200
Minor	Aromatic hydrocarbons	---
Trace	Possibly 2,4-ditert butyl phenol	---

TABLE 23. LRMS REPORT
XAD-2 MODULE WASH
LC FRACTION 7

1. Categories Present

Intensity	Category	MW range
Minor	Aliphatic hydrocarbons, rings or olefins present	41, 43, 55, 57, 69, 71, etc.
Major	HCl^{35} and HCl^{37}	36, 38
Minor	SO and SO_2 (only seen at higher probe temperatures)	48, 64
Moderate	CH_3Cl^{35} and CH_3Cl^{37}	50, 52
	Ph-R cleavage likely from phthalate esters	77, 91, 105
	Possible CH_2=CHPh (NO_2) (present only when sample at 125° C)	148
Major	Phthalate esters	149
Minor	Unassigned-chlorinated	151, 153, 155
Minor	Chlorinated organic	See table 25 for compound F

TABLE 24. LRMS REPORT
XAD-2 MODULE WASH
LC FRACTION 8

1. **Categories Present**

Intensity	Category	MW range
Major	HCl^{35} and HCl^{37}	36, 38
Minor	Phthalate esters (artifacts)	149
	Methvl seloxines (artifacts)	207
Major	Chlorinated hydrocarbon. At least 6 chlorine atoms (for this report this is compound A)	56, 91, 93, 126, 128, 130, 161, 165, 167, 196, 198, 200, 231, 233, 235, 266, 268, 270, 272
Major	Chlorinated hydrocarbon, at least 3 chlorine atoms (for this report this is compound B)	73, 108, 110, 143, 145, 147, 178, 180, 182
Minor	Chlorinated hydrocarbon. Believed to contain at least 8 chlorine atoms (for this report this is compound C)	198, 200, 202, 233, 235, 268, 270, 272, 303, 305, 307, 309, 338, 340, 342, 344
Minor	Chlorinated hydrocarbon, contains at least 9 chlorine atoms (for this report this is compound D)	215, 217, 219, 221, 250, 252, 254, 256, 285, 287, 289, 291 320, 322, 324, 326 328, 355, 357, 359, 390, 392, 394, 396, 398, 400

TABLE 24 (con.)

1. Categories Present

Intensity	Category	MW range
Moderate	Chlorinated hydrocarbon, contains at least 7 chlorine atoms (for this report this is compound E)	224, 226, 228, 259, 261, 263, 265, 294, 296, 298, 300, 302
Moderate	Dichloroethane	63, 65, 98, 100, 102

TABLE 25. LRMS REPORT
XAD-2 CANNISTER WASH
LC FRACTION 7

1. Categories Present

Intensity	Category	MW range
Moderate	Chlorinated organic	See Table 24 for compound A
Minor	Chlorinated organic	See Table 24 for compound C
	Chlorinated organic contains at least 6 chlorine atoms (for this report this is compound F)	116, 118, 120, 122 151, 153, 155, 157, 186, 188, 190, 221, 223, 225, 227, 229, 256, 258, 260
Major	Methylene chloride	50, 52
Trace	Hydrocarbons	41, 43, 55, 57, etc.
Minor	Phthalates	149

TABLE 26. LRMS REPORT
XAD-2 CANNISTER WASH
LC FRACTION 8

1. Categories Present

Intensity	Category	MW Range
Trace	SO_2	64, 48
Minor*	Hydrocarbons	41, 43, 55, 57, etc.
Minor*	Phthalates	149
Major	Chlorinated organic	See table 24 for compound A
Minor	Chlorinated organic	See table 24 for compound B
Major	Chlorinated organic	See table 24 for compound E
Moderate	Chlorinated organic (Peaks caused by this compound more intense at lower sample probe temperatures.)	See table 25 for compound F
	Dichloroethane (Peaks more intense at higher sample probe temperatures.)	63, 65, 98, 100, 102

---

*These peaks may arise from phthalate esters.

TABLE 27. LRMS REPORT
COAL FEED
LC FRACTIONS 6, 7, and 8*

1. Categories Present

Intensity	Category	MW range
	None	Dense peak pattern with a peak at every atomic mass unit (commonly called a pickett fence).
Major	Hydrocarbon	41, 43, 55, 57, 69, 71, 83, 87 are more intense than picketts
Minor	CH_3Cl	50, 52
Major	Phenyl†	77, 91, 105, 119
Major	Alkyl substituted phenols†	107, 108, 121, 122
Moderate	Phthalates	149

*LC 7 has the same composition as LC6 except that the phenols (107, 108, 121, 122) were not noted. LC 8 has the same composition as LC 7 except that a trace of a chlorinated organic, believed to be compound F (see table 25), is seen.

†These patterns are probably related.

STUDY NUMBER 4

DATA SOURCE:	**EFFECT OF A FLYASH CONDITIONING AGENT ON POWER PLANT EMISSIONS** EPA-600/7-76-027
DATA STATUS:	Final Report, October 1976
AUTHOR: CONTRACTOR:	Leslie E. Sparks Industrial Environmental Research Laboratory Office of Energy, Minerals, and Industry U.S. Environmental Protection Agency Research Triangle Park, NC 27711 Program Element No. EHE624

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GENERAL

In electric generating plants, the low-sulfur coal burned to reduce emissions of sulfur gases produces a flyash having a high resistivity, and thus a low collection efficiency by electrostatic precipitation (ESP). The purpose of this study, conducted at Pennsylvania Power and Light Company's Montour Station, was to evaluate the efficiency of a flyash conditioning agent, added to decrease resistivity of the low-sulfur coal's flyash, producing an effluent gas that is low in both sulfur gases and suspended particulates. This flyash conditioning agent, called LPA-402A, was identified as diethylamine sulfate in methanol and water. A secondary goal of the program was to characterize effluent gases during use of LPA-402A. The testing was done in three parts during combustion of: (1) low-sulfur coal + LPA-402A, (2) high-sulfur coal, and (3) low-sulfur coal plus water. Four types of measurements were made: (1) chemical characterization (SO_2, SO_3, NH_3, and organics), (2) flyash resistivity, (3) flyash particle size distribution, and (4) opacity. The data indicated that LPA-402A reduced resistivity of the flyash from 10×10^{10} ohm-cm to 4×10^{10} ohm-cm, but also that ESP efficiency was still too low using low-sulfur coal + LPA-402A to meet emission standards. In addition to this, the LPA-402A may have resulted in increased effluent levels of SO_3, SO_2, and NH_3. Two additional observations drawn from this study were that the SO_3 concentration was not significantly different between the ESP inlet and the ESP outlet and that this electrostatic precipitation system responded slowly to changes in flyash resistivity. This study indicated a need for further work on formulating and testing more suitable flyash conditioning agents.

In summary, average values during the three phases of the test were:

	SO_2 (ppm)	SO_3 (ppm)	NH_3 (ppm)	Flyash resistivity (ohm/cm)
Low-sulfur coal no conditioners (H_2O injection)	663	16	0	9.9×10^{10}
Low-sulfur coal + LPA-402A	879	23	5	4.0×10^{10}
High-sulfur coal no conditioners	1,141	2	0	1.5×10^{10}

GASEOUS GRAB

A special sampling train containing isopropyl alcohol to absorb SO_3 and 3% hydrogen peroxide to absorb SO_2 was used to sample sulfur oxides at the ESP inlet and ESP outlet. Analysis was by the thorin titration method.

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Measurements at both sites were not significantly different. Another specialized train with three impingers containing dilute sulfuric acid was used to sample for ammonia. NH_3 analysis employed a modified Kjeldahl titration of distillate from the H_2SO_4 impinger solutions.

SASS

A SASS train was not employed in this study. The organic sampling apparatus used in the study consisted of a heated probe, heated cyclone and filter, cooling coil, Tenax-GC adsorption module and a Lear-Siegler standard sampling train. Only the Tenax-GC module was taken for analysis. Organic analyses were conducted according to the "Technical Manual for Analysis of Organic Materials in Process Streams." Qualitative analysis of extracts and LC fractions was by FTIR. Specific analysis for diethylnitrosamine, a likely decomposition product of the additive, was by GC-MS, although none was detected (minimum detection limit = 5 ppt). Samples were drawn at the ESP inlet and ESP outlet.

Particle size measurement employed cascade impactors, a Brinks instrument at the ESP inlet and an Andersen instrument at the ESP outlet. Data on particle size distribution from cascade impactors are not readily equated to the Level I data from sequential cyclones, but graphical representations of cascade impactor data at the ESP inlet and outlet are given in the report.

FUGITIVE EMISSIONS

Sampling was not performed in this study.

LIQUIDS AND SLURRIES

Sampling was not performed in this study.

SOLIDS

Sampling was not performed in this study.

TABLE 1. GAS CHROMATOGRAPHY* FOR INORGANIC GASES FLUE GAS PRODUCED FROM INPUT VARIATIONS

Sample	SO_2 ppm	SO_3 ppm	NH_3 ppm
Low-sulfur coal + LPA-402A conditioner	879	28.8	7.9
High-sulfur coal	1,141	23	†
Low-Sulfur coal + water injection	663	16	†

*Sulfur oxides determined by IPA-thorin titration, NH_3 by modified Kjeldahl titrimetric procedure.

†Not detected.

TABLE 2. LC FRACTIONATION*
LOW-SULFUR COAL

	J1 LPA 402A Injected				J2 LPA 402A Injected			
Fraction	TCO	Grav mg	Total	Total	TCO	Grav mg	Total	Total
LC 1		0.23				2.4		
LC 2		1.1				0.44		
LC 3		0.54				0.54		
LC 4		6.8				9.1		
LC 5		0.82				0.84		
LC 6		5.7				6.5		
LC 7		1.4				0.09		
LC 8		0.06				0.03		

	BW1 Water Injected			
Fraction	TCO	Grav mg	Total	Total
LC 1		1.4		
LC 2		3.8		
LC 3		0.47		
LC 4		1.0		
LC 5		0.47		
LC 6		7.7		
LC 7		0.01		
LC 8		0.005		

*Approximately 1,415 l of gas sampled and collected on Tenax-GC column. Pentane extraction for 24 h with one-half of extracted sample fractionated. GC-MS performed on unfractionated half. (Sample weights not available.)

TABLE 3. LC FRACTIONATION* HIGH-SULFUR COAL

	J3 No Injection				J4 No Injection			
Fraction	TCO	Grav mg	Total	Total	TCO	Grav mg	Total	Total
LC 1		0.23				0.18		
LC 2		0.18				0.077		
LC 3		0.23				0.13		
LC 4		6.0				3.9		
LC 5		0.18				0.21		
LC 6		0.48				0.27		
LC 7		0.04				0.03		
LC 8		0.08				0.01		

*Approximately 1,415 l of gas sampled and collected on Tenax-GC column. Pentane extraction for 24 h with one-half of extract sample fractionated. GC-MS performed on unfractionated half. (Sample weights not available.)

TABLE 4. IR REPORT
SAMPLE: LOW-SULFUR COAL + LPA 402A CONDITIONER

LC	J1	J2
1	Silicone, aromatic hydrocarbon (multiple or fused ring)	Silicone, aromatic hydrocarbon
2	Same as LC 1	Silicone, aromatic hydrocarbon, ester (trace)
3	Silicone, aromatic hydrocarbon, aliphatic ester	Silicone, ester (trace), aromatic ketone or quinone
4	Aromatic ketone or quinone	Aromatic ketone or quinone
5	Aromatic ketone or quinone, aliphatic ketones	Aromatic ketone or quinone, aliphatic ketones
6	Carboxylic acid, aliphatic ketones	Carboxylic acid, aliphatic ketones
7	Carboxylic acid (soaps), alcohol (aliphatic, polyalcohol or alkanol amine)	Carboxylic acid salt (soap)
8	*	*

*Not reported.

TABLE 5. IR REPORT
SAMPLE: LOW-SULFUR COAL + WATER INJECTION

LC	BW1
1	Silicone trace
2	Aromatic ketone or quinone
3	Same as LC 2
4	Aromatic ketone or quinone, substituted phenol
5	Same as LC 4
6	Highly substituted phenol (possible polyhydroxy compound)
7	*
8	*

*Not reported.

TABLE 6. IR REPORT
SAMPLE: HIGH-SULFUR COAL

LC	J3	J4
1	Silicone trace	Silicone
2	Silicone (trace), aromatic hydrocarbon (trace)	Silicone, aromatic hydrocarbon
3	Aromatic ketone or quinone	Aromatic ketone or quinone
4	Same as LC 3	Same as LC 3
5	Aromatic ketone or quinone, 2nd aromatic ketone/quinone	Aromatic ketone or quinone, 2nd aromatic ketone/quinone
6	Aliphatic ketone	Aliphatic ketone, 2nd aromatic ketone/quinone
7	*	*
8	*	*

*Not reported.

STUDY NUMBER 5

DATA SOURCE:

ORGANIC AND SULFATE SAMPLING ANALYSIS AT COLBERT STEAM PLANT

GCA-TR-76-36-G

DATA STATUS: Draft Final Report, October 1976

AUTHORS: Robert M. Bradway and Donald F. Durocher

CONTRACTOR:
GCA Corporation
GCA/Technology Division
Bedford, Massachusetts 01730
Contract No. 68-02-1316, Task 21

PROJECT OFFICER:
Ronald A. Venezia
Chemical Processes Branch
Industrial Environmental Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

GENERAL

A study was performed by GCA at TVA's coal-fired Colbert Steam Plant for Level I organics plus sulfates, PCB's, and benzo(a)pyrene (B(a)P). Particulate loading was performed to assess the efficiency of the electrostatic precipitator (ESP) which was tested at a particle removal efficiency of 99.58% with an average outlet concentration of 0.0131 grains/scf. Concurrent additional testing at this same site was conducted by TVA, and results will be published separately by TVA. Sampling took place during nearly full load conditions and concentrated on chemically characterizing the emissions from Colbert's Unit One, a 185-MW boiler with dual exhaust passages (herein designated ducts A and B) to an ESP. Two tests (1 and 2) were run on the duct A ESP outlet, and test 3 was run on the duct A ESP inlet.

GASEOUS GRAB

The Level I methodology was not employed. Federal Register methods with a RAC 2343 source sampling train were used to determine CO_2, O_2, CO, and H_2O, as well as other parameters.

SASS

A SASS train was not used in this study. Particulate-containing gaseous streams, specifically the ESP outlet duct from Unit One, were sampled with a RAC 2343 sampling system, a standard sampling system for Federal Register methods 2 through 8 (excluding method 7), and modified to include a Tenax-GC adsorbent module between the filter and impingers. Since no particle-sizing cyclone system was employed, sampling velocity was adjusted with changes in effluent stream velocity. Part of the filter from run 2 was methylene chloride extracted in a Soxhlet apparatus, and part of the Tenax was, likewise, pentane extracted. A portion of the run 2 impinger wash (200 ml) was extracted in a separatory funnel with methylene chloride. The following table from the text summarizes the organic analysis scheme.

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TABLE 1. PROPERTIES OF EXTRACT AND COMBINED SAMPLE

Sample	Tenax	Filter	Probe wash
Sample size	4.2303 g	0.1104 g	200 ml
Extract medium	Pentane	Methylene chloride	Methylene chloride
Extraction time	26 h	26 h	20 min
Extract color	Yellow	Straw yellow	Colorless
Weight of extract	0.8737 g	0.7045 g	1.7025 g
Weight of extract used in combined sample	0.2355 g	0.2232 g	0.5574 g

The three extracts were combined, and LC fractionation was run according to specified Level I procedures. IR analysis was run on each LC fraction, but no LRMS analysis was run.

B(a)P analysis was run on a portion of filter extract from run 2 using a TLC separation with UV spectrophotometric quantification. Sulfate analysis was run on an aqueous filter extract using the spectrophotometric barium chloranilate method.

FUGITIVE EMISSIONS

Not sampled in this study.

LIQUIDS AND SLURRIES

Not sampled in this study.

SOLIDS

Not sampled in this study.

TABLE 2. ANION ANALYSIS FOR PARTICULATES

Sample	Cl	F	SO_4*	SO_3	NO_3	NO_2
Test 2 (ESP outlet)			5.19 $\mu g/m^3$			

*Sulfate analysis was by the spectrophotometric method (barium chloranilate method). The sample was extracted for 3 h with 25 ml hot water in a Soxhlet apparatus. Appropriate sample aliquots were mixed with 25 mg of barium chloranilate and isopropanol, shaken, and centrifuged. The UV absorbance of the samples over the wavelength range 290 to 330 nm was recorded using a P and E Model 202 UV-VIS spectrophotometer. Absorbance values were determined at 310 nm for blank and calibration standards, and a concentration versus intensity curve was prepared.

TABLE 3. GAS CHROMATOGRAPHY FOR INORGANIC GASES

Sample	CO%	O_2%	CO_2%
Test 1 (8/10/76 at ESP outlet)	0.0	6.5	11.6
Test 2 (8/11/76 at ESP outlet)	0.10	7.1	11.9
Test 3 (8/12/76 at ESP inlet	0.20	6.3	12.9

TABLE 4. LC FRACTIONATION COMBINED EXTRACTS FROM TENAX-GC CARTRIDGE, FILTER, AND PROBE WASH

Fraction	TCO	Grav µg	Total	Total
LC 1		633		
LC 2		59		
LC 3		166		
LC 4		306		
LC 5		247		
LC 6		872		
LC 7		1,190		
LC 8		58,118		

TABLE 5. IR REPORT
COMBINED EXTRACTS FROM TENAX-GC CARTRIDGE, FILTER, AND PROBE WASH

LC	Wave number (cm^{-1})	Intensities	Assignment	Comments
1	2,850-2,940		Aliphatic hydrocarbons	
	1,470		Aliphatic hydrocarbons	
	1,375		Aliphatic hydrocarbons	
2	Wide broad bands across most of spectrum		Not identified	Aromatic hydrocarbons are not major constituent (no band at 3,050 cm^{-1})
3	2,850-2,970		Alkyl groups	
	Broad bands between 1,300-2,000. Sharp band at 1,270		Possible carbonyl compound Possible carbonyl compound Possible carbonyl compound	
4	Broad band at 3,450 cm^{-1} Sharp band 2,850-2,970		Possible alcohols or phenols	
5	3,050	Weak	Aromatics	
	Broad band 3,400		Mixture of carbonyl compounds	
	Sharp band 1,650		Mixture of carbonyl compounds	
	Bands 800-1,300	Weak	Mixture of carbonyl compounds	
	700, 755, 780	Strong	Substituted aromatic groups	
6	1,180	Strong	Not identified	
	Bands below 1,300		Organic/inorganic sulfurs	Yellow
	850	Strong	Substitute aromatics	
7	3,400	Strong		
	Sharp bands at 1,590, 1,725, 1,060, 1,080 and 1,120		Carbonyl compounds and alcohols	
8	2,400, 825, 1,000, 1,100		Amines, imines, or amino acids sulfonic acid salts	Conflicting indications of water present. Strong bands at 3,200 and 1,640; no band at 2,100.

STUDY NUMBER 6

DATA SOURCE: **A NEW ENERGY SOURCE**

AUTHORS: Karl J. Bombaugh

CONTRACTOR:
Radian Corporation
8500 Shoal Creek Blvd
P.O. Box 9948
Austin, Texas 78766
R.C. #200-143
DCN 77-200-143-14

PROJECT OFFICER:
Bill Rhodes
Industrial Environmental Research Laboratory
Fuel Process Branch
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

TABLE 1. SPARK SOURCE MASS SPECTROSCOPY
SITE A--SAMPLE 2
(ppmw)

U		Dy		Rh		Cr	3
Th		Tb		Ru		V	0.8
Bi		Gd		Mo	1	Ti	29
Pb	10	Eu		Nb		Sc	0.7
Tl		Sm		Zr	0.7	Ca	630
Hg	0.12*	Nd	0.6	Y	≤0.2	K	100
Au		Pr	0.3	Sr	10	Cl	6
Pt		Ce	0.5	Rb	0.2	S	520
Ir		La	0.6	Br	2	P	17
Os		Ba	27.0	Se	3	Si	170
Re		Cs	'0.1	As	4	Al	25
W		I	1	Ge	1	Mg	23
Ta		Te		Ga	8	Na	71
Hf		Sb	0.8	Zn	7	F	≈22
Lu		Sn	0.9	Cu	3	B	19
Yb		In		Ni	5	Be	0.1
Tm		Cd		Co†	5	Li	4
Er		Ag		Fe	120		
Ho		Pd		Mn	0.9		

*Hg data by flameless AAS.
†Heterogeneous.
No data indicates concentration < 0.2 ppmw.

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY
SITE A--SAMPLE 3
(μg/l)

U		Dy		Rh		Cr	0.03
Th		Tb		Ru		V	
Bi		Gd		Mo	0.06	Ti	0.05
Pb	0.04	Eu		Nb		Sc	≤0.006
Tl		Sm		Zr	0.01	Ca	‡
Hg	0.007*	Nd	<0.01	Y		K	‡
Au		Pr	0.005	Sr	0.2	Cl	0.3
Pt		Ce	0.01	Rb	0.03	S	‡
Ir		La	≤0.01	Br	0.2	P	‡
Os		Ba	0.1	Se	4.0	Si	7
Re		Cs	'1	As	0.2	Al	1
W		I	0.5	Ge	≤0.02	Mg	2
Ta		Te		Ga	0.006	Na	‡
Hf		Sb	0.1	Zn	0.07	F	≈2
Lu		Sn	0.02	Cu	0.1	B	2
Yb		In	†	Ni	0.1	Be	
Tm		Cd	≤0.02	Co	≤0.008	Li	0.2
Er		Ag		Fe	3.0		
Ho		Pd		Mn	0.03		

*Hg data by flameless AAS.
†Internal standard.
‡Major component.
No data indicates concentration ≤ 0.004, μg/l.

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TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
SITE A--SAMPLE 4
(ppmw)

U	56	Dy	17	Rh		Cr	510
Th	86	Tb	4	Ru		V	*
Bi	0.4	Gd	10	Mo	22	Ti	*
Pb	7	Eu	5	Nb	82	Sc	29
Tl	0.5	Sm	28	Zr	430	Ca	*
Hg		Nd	56	Y	260	K	*
Au		Pr	42	Sr	*	Cl	230
Pt		Ce	260	Rb	120	S	250
Ir		La	280	Br	12	P	*
Os		Ba	*	Se	20	Si	*
Re	0.3	Cs	10	As	4	Al	*
W	10	I	0.3	Ge	4	Mg	*
Ta	2	Te		Ga	66	Na	*
Hf	10	Sb	1	Zn	26	F	≈56
Lu	2	Sn	4	Cu	540	B	130
Yb	12	In	†	Ni	120	Be	22
Tm	1	Cd	3	Co	61	Li	190
Er	8	Ag	≤0.3	Fe	*		
Ho	11	Pd		Mn	680		

*Major component.
†Internal standard.
No data indicates concentration <0.2 ppmw.

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
SITE A--SAMPLE 5
(ppmw)

U		Dy		Rh		Cr	90
Th		Tb	9	Ru		V	100
Bi	2	Gd	2	Mo	14	Ti	‡
Pb	60	Eu	1	Nb	12	Sc	12
Tl		Sm	9	Zr	80	Ca	‡
Hg	0.01*	Nd	21	Y	70	K	‡
Au		Pr	5	Sr	340	Cl	720
Pt		Ce	45	Rb	33	S	‡
Ir		La	45	Br	20	P	‡
Os		Ba	460	Se	24	Si	‡
Re		Cs	1	As	27	Al	‡
W		I	4	Ge	5	Mg	‡
Ta		Te	9	Ga	130	Na	‡
Hf		Sb	8	Zn	85	F	≅720
Lu		Sn	2	Cu	130	B	70
Yb		In	†	Ni	30	Be	6
Tm		Cd	<2	Co	16	Li	27
Er		Ag	3	Fe	‡		
Ho		Pd		Mn	120		

*Hg data by flameless AAS.
†Internal standard.
‡Major component.
No data indicates concentration < 0.2 ppmw.

TABLE 5. ATOMIC ABSORPTION (AA)*--WET CHEMICAL METHODS
SITE A

Sample	Hg	Sb†	As†
2 (ppm)	0.12	0.8	4
3 (μg/1)	.007	0.1	0.2
4 (ppmw)	----	1	4
5 (ppmw)	0.01	8	27

*AA used to determine Hg.

†SSMS used to determine Sb and As.

TABLE 6. LC FRACTIONATION--SITE A

	Sample 2				Sample 3			
Fraction	TCO	Grav mg/g	Total	Total	TCO	Grav mg/l	Total	Total
LC 1		1.49				0.3		
LC 2		10.95				1.8		
LC 3		96.80				5.0		
LC 4		47.00				17.0		
LC 5		41.03				163.0		
LC 6		293.37				1,655.0		
LC 7		27.88				69.0		
LC 8		41.63				186.0		

	Sample 4				Sample 5			
Fraction	TCO	Grav	Total	Total	TCO	Grav ppm	Total	Total
LC 1		Extract of 50.3 g				109		
LC 2		sample yielded only				18		
LC 3		0.89 mg of organics, so				1		
LC 4		no LC separation was				3		
LC 5		performed.				3		
LC 6						113		
LC 7						19		
LC 8						44		

TABLE 7. IR REPORT: SAMPLES FROM SITE A

LC	Sample 2	Sample 3
1	Aliphatic hydrocarbons $(-CH_2-)_{>4}$, some branched	Aliphatic hydrocarbons
2	Substituted aromatic with alkyl and branched alkyl substituents	Alkyl-aryl hydrocarbons
3	Substituted aromatic with alkyl and branched alkyl substituents	Alkyl-aryl hydrocarbons, trace carbonyl, possible polycyclics or multisubstituted aromatic
4	Same as above, plus NH stretch	Alkyl-aryl hydrocarbons, possible polycyclics, -OH present (possibly atmospheric moisture)
5	Same as above, but probably OH stretch, trace C = 0	Phenol + alkyl/diakyl phenol
6	Bonded OH and NH stretch	Principally phenol + other phenols
7	Primary OH, water of hydration, CO, ether, ester (acetate), split carbonyl (possibly acid)	Phenols
8	Split C = 0 (acid and ester), water of hydration, possible ester (acetate), aliphatic, methyl group	Alkyl-aryl CH stretch, OH, C = 0, methyl bending vibration, primary OH, possible inorganic sulfur + other ionic compounds

TABLE 7 (con.)

LC	Sample 4	Sample 5
1		Paraffinic hydrocarbons, considerable branching
2		Paraffinic hydrocarbons, trace substituted aromatics
3&4		Split carbonyl (formate or butryate), methyl, isopropyl and tributyl branching, primary alcohols
5	*No separation was performed due to low* organic concentration in sample. IR indicates presence of aliphatic hydrocarbons and possibly ester or acetate	Aromatics, carbonyl, aliphatic hydrocarbons
6		Split carbonyl; methyl, isopropyl and tributyl branching secondary alcohols; ester possible 5-C ring lactone; branched cyclic alcohol
7		No assignment made
8		Not reported

TABLE 8. GAS CHROMATOGRAPHY FOR C_7-C_{17}
SITE A

Sample	Range	Volatile weight	No. of peaks	Gravimetric nonvolatile weight	Total organic
#3 (ppm)	GC7 90 - 110	260*	----	----	
	GC8 110 - 140	0			
	GC9 140 - 160	0			
	GC10 160 - 180	2,544			
	GC11 180 - 200	2,766			
	GC12 200 - 220	917			
	GC 13 and greater	800			
#2 (mg/g of sample)	GC7 90 - 110	3.0			
	GC8 110 - 140	0			
	GC9 140 - 160	0			
	GC10 160 - 180	18.2			
	GC11 180 - 200	56.7			
	GC12 200 - 220	72.5			
	GC13 and greater	307.5			
#5 (mg/g of sample)	GC7 90 - 110	2.2	No components detected in C_7-C_{12} range (except the one peak), nor in range C_1-C_6; therefore, the peak reported here originated from the extracting solvent.		
	GC8 110 - 140				
	GC9 140 - 160				
	GC10 160 - 180				
	GC11 180 - 200				
	GC12 200 - 220				

*260 may have originated from extraction solvent since GC8 and GC9 detect nothing.

STUDY NUMBER 7

DATA SOURCE:

ANALYSIS OF COKE OVEN QUENCH TOWER EMISSIONS

DATA STATUS:

Letter, April 1977

TO:

Ms. Diane K. Sommerer
Director of Environmental Sciences
York Research Corporation
1 Research Drive
Stamford, Connecticut 06906

FROM:

Peter W. Jones
Battelle-Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

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GENERAL

Fifteen samples from a coke quenching operation were collected for Level 1 analyses and POM analysis by GC-MS. Details of sampling procedures and experimental design were not given in the letter.

Level 1 analysis for organics was performed on ten samples according to the Technical Manual on Analysis of Organic Materials in Process Streams, and data formatted for organics includes FTIR and gravimetric analyses. SSMS elemental analysis was performed on four samples.

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TABLE 1. SPARK SOURCE MASS SPECTROSCOPY
FILTER BLANK
(ppmw)

U	<2	Dy	<0.5	Rh	<0.5	Cr	10
Th	<0.1	Tb	<0.2	Ru	<0.3	V	0.5
Bi	<0.1	Gd	<0.5	Mo	2	Ti	20
Pb	<10*	Eu	<0.3	Nb	0.5	Sc	2
Tl	<0.1	Sm	<1	Zr	50	Ca	~2%
Hg	<0.3	Nd	<0.5	Y	1	K	5,000
Au	<0.1	Pr	<0.5	Sr	100	Cl	1,000
Pt	<0.3	Ce	0.2	Rb	5	S	500
Ir	<0.2	La	<0.5	Br	20	P	5
Os	<0.2	Ba	2,000	Se	<50*	Si	~20%
Re	<0.2	Cs	<1	As	≤5*	Al	~2%
W	<0.3	I	<50	Ge	<1	Mg	~3%
Ta	<0.2	Te	<0.2	Ga	<1	Na	~10%
Hf	<0.5	Sb	1	Zn	30	F	300
Lu	<0.2	Sn	2	Cu	50	B	2,000
Yb	<2	In	<2*	Ni	50	Be	0.03
Tm	<0.2	Cd	<0.3	Co	3	Li	20
Er	<0.3	Ag	<2	Fe	1,000		
Ho	<0.1	Pd	<2	Mn	30		

*Memory from previous sample.

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY
FILTER COMPOSITE SAMPLE--
NOT BLANK CORRECTED
(ppmw)

U	<2	Dy	<1	Rh	<0.5	Cr	50
Th	<0.1	Tb	<0.3	Ru	<0.5	V	2
Bi	<0.1	Gd	<1	Mo	5	Ti	100
Pb	<0.2*	Eu	<1	Nb	2	Sc	2
Tl	<0.3	Sm	<1	Zr	100	Ca	~5%
Hg	<0.3	Nd	<3	Y	5	K	~1%
Au	<0.1	Pr	1	Sr	1,000	Cl	~2%
Pt	<0.5	Ce	10	Rb	10	S	2,000
Ir	<0.3	La	5	Br	200	P	30
Os	<0.3	Ba	~2%	Se	<50*	Si	~20%
Re	<0.2	Cs	<1	As	<5*	Al	~2%
W	<0.3	I	<2	Ge	<3	Mg	~3%
Ta	<1	Te	<0.5	Ga	<1	Na	~10%
Hf	3	Sb	5	Zn	10	F	500
Lu	<1	Sn	1	Cu	100	B	2,000
Yb	<2	In	<2%	Ni	100	Be	0.1
Tm	<0.5	Cd	<1	Co	5	Li	50
Er	<0.5	Ag	<1	Fe	1,000		
Ho	<0.5	Pd	<2	Mn	30		

*Memory from previous sample.

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
FILTER COMPOSITE--BLANK CORRECTED
(ppmw)

U		Dy		Rh		Cr	40
Th		Tb		Ru		V	1.5
Bi		Gd		Mo	3	Ti	80
Pb		Eu		Nb	1.5	Sc	
Tl		Sm		Zr	50	Ca	~3%
Hg		Nd		Y	4	K	5,000
Au		Pr	1	Sr	900	Cl	19,000
Pt		Ce	9.8	Rb	5	S	1,500
Ir		La	5	Br	180	P	25
Os		Ba	18,000	Se		Si	
Re		Cs		As		Al	
W		I		Ge		Mg	
Ta		Te		Ga		Na	
Hf		Sb	4	Zn		F	200
Lu	3	Sn		Cu	50	B	
Yb		In		Ni	50	Be	0.07
Tm		Cd		Co	2	Li	30
Er		Ag		Fe			
Ho		Pd		Mn			

Blank data spaces indicate sample valves ≤ blank values.

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
INCANDESCENT COKE
(ppmw)

U	<0.5	Dy	<1	Rh	<0.3	Cr	100
Th	<0.5	Tb	<0.3	Ru	<2	V	100
Bi	<0.3	Gd	<1	Mo	5	Ti	3,000
Pb	≤5*	Eu	<1	Nb	5	Sc	20
Tl	<1	Sm	<1	Zr	30	Ca	2,000
Hg	<1	Nd	10	Y	10	K	1,000
Au	<0.3	Pr	2	Sr	300	Cl	100
Pt	<1	Ce	5	Rb	30	S	500
Ir	<1	La	10	Br	5	P	100
Os	<1	Ba	200	Se	<20*	Si	~2%
Re	<0.5	Cs	1	As	≤300*	Al	~2%
W	<1	I	0.2	Ge	<2	Mg	~1%
Ta	<2	Te	<1	Ga	30	Na	2,000
Hf	<1	Sb	1	Zn	5	F	3
Lu	<0.5	Sn	5	Cu	100	B	50
Yb	<1	In	<1*	Ni	20	Be	10
Tm	<3	Cd	<1	Co	50	Li	100
Er	<1	Ag	<1	Fe	~1%		
Ho	<0.5	Pd	<2	Mn	200		

*Memory from previous sample.

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY
QUENCHED CLEAN COKE
(ppmw)

U	<0.5	Dy	<1	Rh	<0.3	Cr	100
Th	<0.5	Tb	<0.3	Ru	<2	V	100
Bi	<0.3	Gd	<1	Mo	5	Ti	5,000
Pb	≤30*	Eu	<1	Nb	20	Sc	30
Tl	<1	Sm	<1	Zr	100	Ca	3,000
Hg	<2	Nd	20	Y	30	K	2,000
Au	<0.3	Pr	10	Sr	300	Cl	300
Pt	<1	Ce	20	Rb	100	S	2,000
Ir	<1	La	50	Br	30	P	300
Os	<1	Ba	500	Se	<30*	Si	~10%
Re	<0.5	Cs	3	As	≤300*	Al	~5%
W	<1	I	2	Ge	<10	Mg	~1%
Ta	<3	Te	<1	Ga	30	Na	3,000
Hf	<2	Sb	3	Zn	50	F	3
Lu	<0.3	Sn	100	Cu	200	B	50
Yb	<1	In	<2*	Ni	100	Be	5
Tm	<3	Cd	<2	Co	50	Li	100
Er	<1	Ag	<1	Fe	~5%		
Ho	<0.5	Pd	<1	Mn	1,000		

*Memory from previous sample.

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY
QUENCHED DIRTY COKE
(ppmw)

U	<0.5	Dy	<3	Rh	<2	Cr	100
Th	<0.5	Tb	<0.3	Ru	<3	V	100
Bi	<0.3	Gd	<1	Mo	20	Ti	5,000
Pb	≤200*	Eu	<1	Nb	10	Sc	20
Tl	<1	Sm	<1	Zr	100	Ca	5,000
Hg	<2	Nd	30	Y	100	K	5,000
Au	<0.3	Pr	10	Sr	3,000	Cl	500
Pt	<1	Ce	100	Rb	100	S	2,000
Ir	<1	La	50	Br	50	P	500
Os	<1	Ba	2,000	Se	<20*	Si	~5%
Re	<0.5	Cs	20	As	≤300*	Al	~5%
W	<1	I	10	Ge	<10	Mg	~1%
Ta	<3	Te	<1	Ga	30	Na	~1%
Hf	<2	Sb	3	Zn	100	F	10
Lu	<1	Sn	~1%	Cu	200	B	50
Yb	<1	In	<10*	Ni	300	Be	20
Tm	<3	Cd	<3	Co	50	Li	200
Er	<2	Ag	<0.5	Fe	~5%		
Ho	<1	Pd	<2	Mn	500		

*Memory from previous sample.

TABLE 7. LC FRACTIONATION

	Coke-quenched, clean				Coke-quenched, dirty			
Fraction	TCO	Grav mg	Total	Total	TCO	Grav mg	Total	Total
LC 1		0.35				0.79		
LC 2		0.030				0.022		
LC 3		0.068				0.038		
LC 4		0.049				0.073		
LC 5		0.069				0.12		
LC 6		0.068				0.042		
LC 7		0.11				0.19		
LC 8		0.090				0.038		

	Coke-quenched, clean			
Fraction	TCO	Grav mg	Total	Total
LC 1		0.14		
LC 2		0.036		
LC 3		11.6		
LC 4		1.38		
LC 5		0.24		
LC 6		0.062		
LC 7		1.52		
LC 8		0.43		

TABLE 7 (con.)

	Dirty water inlet				Dirty water return			
Fraction	TCO	Grav mg	Total	Total	TCO	Grav mg	Total	Total
LC 1		0.13				0.09		
LC 2		0.23				0.054		
LC 3		2.54				1.57		
LC 4		7.74				1.58		
LC 5		12.5				21.0		
LC 6		1.78				0.9		
LC 7		12.6				11.03		
LC 8		0.28				0.42		

	Dirty water makeup liquor			
Fraction	TCO	Grav mg	Total	Total
LC 1		0.12		
LC 2		0.044		
LC 3		2.8		
LC 4		6.1		
LC 5		13.1		
LC 6		2.28		
LC 7		7.45		
LC 8		0.26		

TABLE 7 (con.)

	Clean water samples				Clean water test 11A			
Fraction	TCO	Grav mg	Total	Total	TCO	Grav mg	Total	Total
LC 1		0.90				2.1		
LC 2		0.19				203		
LC 3		0.57				271		
LC 4		0.37				88		
LC 5		2.72				9.3		
LC 6		0.23				2.96		
LC 7		2.95				22.9		
LC 8		0.22				8.51		

	Dirty water test 16				Test 13			
Fraction	TCO	Grav mg	Total	Total	TCO	Grav mg	Total	Total
LC 1		2.7				2.8		
LC 2		0.31				422		
LC 3		44				335		
LC 4		3.4				34		
LC 5		1.58				11.5		
LC 6		4.77				3.25		
LC 7		13.3				42.0		
LC 8		6.2				10.8		

TABLE 8. IR REPORT
SAMPLE: TEST 13

Fraction	Assignments (comments)
LC 1	Aliphatic HC, aromatic HC, silicone grease
LC 2	Aliphatic HC, phthalate(s)*
LC 3	Phthalate(s)
LC 4	Phthalate(s)
LC 5	Phthalate(s), carboxylic acid
LC 6	Phthalate(s), carboxylic acid
LC 7	Similar to acrylate polymer
LC 8	Crystalline hydrocarbon structure, ester†

*These phthalates as well as those in the rest of the set seem to be composed of changing amounts of at least three phthalates. The most likely candidates are:
  (a) Butyl benzyl phthalate
  (b) Dimethyl cyclohexyl phthalate
  (c) Mixed alcohol phthalate(s).
Note that some of the fractions such as 16A - 3,4 and 13A - 3,4 had so much of the phthalates that we had to run the spectra as liquid films rather than solvent cast dry films.

†Surprising to find an ester in fraction 8, could be introduced during separation.

TABLE 9. IR REPORT
SAMPLE: CLEAN WATER TEST 11A

Fraction	Assignments (comments)
LC 1	Aliphatic HC, silicone
LC 2	Silicone, phthalates
LC 3	Phthalates
LC 4	Phthalates
LC 5	Phthalates, aldehyde (aliphatic + carboxylic acid +?)
LC 6	Phthalates, aldehyde + acid +?
LC 7	Phthalates, much polar material, possible acrylates plus other esters
LC 8	Same as fraction 7 without phthalate

TABLE 10. IR REPORT
SAMPLE: COKE ACETONE BLANK

Fraction	Assignments (comments)
LC 1	Polyethylene*, silicone grease
LC 2	Normal[†] amounts of aliphatic and aromatic hydrocarbons, ester
LC 3	Phthalate(s)[‡], polyethylene
LC 4	Phthalate(s)
LC 5	Phthalate(s), ester, polyethylene
LC 6	Phthalate(s)
LC 7	Aliphatic ester IV
LC 8	Aliphatic ester IV

*"Polyethylene" does not exclude the possibility of a long chain hydrocarbon(s).
[†] Normal means similar in levels to that seen in the BCL tenax washed blanks.
[‡] Phthalates are the most common spectral feature in these series.
"Phthalate" may indicate more than one phthalate, i.e., a mix of phthalates.

TABLE 11. IR REPORT
SAMPLE: COKE-QUENCHED CLEAN

Fraction	Assignments (comments)
LC 1	Normal aliphatic hydrocarbons
LC 2	Normal aromatic ester I
LC 3	Hydrocarbon, ester I (aromatic), phthalate I
LC 4	Phthalate I
LC 5	Phthalate
LC 6	Phthalate
LC 7	Phthalate, aliphatic ketone
LC 8	Normal polar materials

TABLE 12. IR REPORT
SAMPLE: COKE-QUENCHED DIRTY

Fraction	Assignments (comments)
LC 1	Aliphatic hydrocarbon, silicone grease
LC 2	Aliphatic hydrocarbon, silicone grease
LC 3	Phthalate I
LC 4	Phthalate I
LC 5	Phthalate II
LC 6	Phthalate II, possible ester
LC 7	Phthalate II, aliphatic ketone
LC 8	Normal polar materials

TABLE 13. IR REPORT
SAMPLE: DIRTY WATER INLET

Fraction	Assignments (comments)
LC 1	Aromatic hydrocarbon, aliphatic hydrocarbon
LC 2	Probably condensed ring aromatic*
LC 3	Carbazole and/or indole types
LC 4	Phenols
LC 5	Phenols, nitrile or N-H compound
LC 6	Phenols, nitrile or N-H compound
LC 7	Carboxylic acid or possible quinone
LC 8	Carboxylic acid or possible quninone, possible amine HCl salt

*Fraction 2 is most likely a condensed ring aromatic, benzopyrene(s) being a definite possibility.

TABLE 14. IR REPORT
SAMPLE: DIRTY WATER MAKEUP LIQUOR

Fraction	Assignments (comments)
LC 1	Aliphatic hydrocarbon
LC 2	Trace ester (normal levels)
LC 3	Carbazole and/or indole types
LC 4	Phenols
LC 5	Phenols
LC 6	Phenols
LC 7	Conjugated aromatic carboxylic acid and other C-O
LC 8	Similar to fraction 7 but less of acid structure

TABLE 15. IR REPORT
SAMPLE: CLEAN WATER SAMPLES

Fraction	Assignments (comments)
LC 1	Aliphatic hydrocarbon
LC 2	Aromatic ester
LC 3	Aromatic ester with alcohol group as well*
LC 4	Similar to fraction 3 but more aromatic nature
LC 5	Phenols
LC 6	Phenols, nitrile
LC 7	Nitrile, aromatic carboxylic acids
LC 8	Aromatic carboxylic acids, ketone, probably aromatic

*Note that this could be an aliphatic alcohol and an aromatic ester, or an aromatic ester with an OH on a side chain of the aromatic ring, or be directly on the ring (a phenol).

TABLE 16. IR REPORT
SAMPLE: DIRTY WATER RETURN

Fraction	Assignments (comments)
LC 1	Aliphatic hydrocarbon (normal levels)
LC 2	Trace ester (normal)
LC 3	Probable carbazole*, carbazole indole types
LC 4	Phenols, other C-O compounds
LC 5	Phenols
LC 6	Phenols, cresol(?), nitrile
LC 7	Carboxylic acid aromatic, possible $C\equiv N$ compound
LC 8	Carboxylic acid aromatic, amine HCl salt

*We have never seen this in a Level 1 before.

TABLE 17. IR REPORT
SAMPLE: DIRTY WATER TEST 16

Fraction	Assignments (comments)
LC 1	Aliphatic hydrocarbon, silicone grease
LC 2	Phthalate, silicone grease
LC 3	Phthalate(s)
LC 4	Phthalate(s)
LC 5	Phthalate(s), ketone, carboxylic acid
LC 6	Phthalate(s), ketone, carboxylic acid
LC 7	Possible isothiocyanate, carboxylic acid
LC 8	Possible isothiocyanate, possible phosphine

STUDY NUMBER 8

DATA SOURCE:

SAMPLING AND ANALYSIS OF COKE OVEN DOOR EMISSIONS

EPA-600/2-77-213

DATA STATUS: Final Report, October 1977

AUTHORS: R. E. Barrett, W. L. Margard,
J. B. Purdy, and P. E. Strup

CONTRACTOR: Battelle-Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201
Contract No. 68-02-1409, Task 34
Program Element No. 1AB604C

PROJECT OFFICER: Robert C. McCrillis
Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

A specially constructed hood of black metal with cooling vents, fitted over the entire front of an oven door, was used to sample emissions from leaks in coke oven doors at the Republic Steel Corporation's plant in Youngstown, Ohio. During sampling, metered air was blown in through the bottom of the hood and metered air was drawn off at the top so that a zero pressure change was maintained across the face of the door; in this manner, measurements of the amount of gaseous emissions could be made. Figure 1 from the text shows the sampling setup and figure 2 schematically shows the sampling apparatus. Two coking cycles of about 16 hours were sampled on 3/31/76 (run 1) and 4/1-2/76 (run 2). Since sampling problems were encountered in run 1 and since run 2 indicated much greater quantities of emissions than run 1, run 2 was chosen for detailed Level I analysis. Results indicate that a large percent of the emissions occur during the first hour of coking, with variations in this percent possibly dependent on tightness of the door seal. Large amounts of POM are emitted with benzopyrene emissions at the top of the list. Data indicate particulates from filters are heavily laden with mutagens. Ames assay found mutagens in all samples.

GASEOUS GRAB

With a coking cycle of approximately 16 hours and an anticipated high initial emissions level, six samples were taken at startup +0.5, +1.5, +3.0, +6.0, +10.0, and +13.0 hours. The evacuated glass bulb sample was withdrawn from the sampling apparatus (see description in Fugitive Emissions Section) downstream from a Tenax-GC adsorber cartridge.

SASS

A SASS train was not used in this study.

FUGITIVE EMISSIONS

The blower from the specially constructed sampling hood directed an effluent stream through an 8 in. x 10 in. high volume filter. A smaller line of Tygon tubing connected the effluent stream, prior to the filter, to an adsorber capsule containing Tenax-GC. Another section of Tygon tubing directed the effluent stream from the adsorber capsule to an evacuated gas bulb sampler.

After being weighed, the Hi-Vol filters were divided into quarters for organic and inorganic analysis and bioassay. Filter portions for organic analysis were Soxhlet extracted sequentially with methylene chloride and methanol until the solvent around the Soxhlet thimble remained clear. Extracts were combined and reduced in volume. The Tenax-GC adsorbent was extracted for 24 hours with pentane. Extracts were evaporated to dryness. All the sample from Tenax-GC extraction and 1/600th of the sample from

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filter extraction were subjected to LC fractionation per "The Technical Manual for Analysis of Organic Materials in Process Streams," EPA 3/76. A Fourier Transform Infrared System (FTIR) was used for IR analysis. Representative filter and adsorber samples from each LC class fraction were selected for GC-MS analysis (rather than LRMS).

Filter portions for inorganic analysis were extracted in hot aqua regia, and the resulting extract was dried, then mixed in a ratio of 3 parts sample to 1 part graphite to make an SSMS electrode. Wet chemical-atomic absorption analyses for Hg, Sb, and As were not conducted.

Sampling periods and identifications follow:

IDENTIFICATION OF TEST NUMBER 2 SAMPLES

Sampling time	Filter nos.	BCL code	Adsorber nos.	BCL code
1303-1411	119-131	A1F	2.1, 2.2	A1
1414-1506	132-136	A2F	2.3, 2.4	A2
1506-1708	137-147	A3F	2.5, 2.6	A3
1708-2105	148-155	A4F	2.7, 2.8	A4
2105-0215	156-161	A5F	2.9, 2.10 2.11	A5
Compressor air supply (blank)	---	---	TP.4	A6

LIQUIDS AND SLURRIES

No liquids or slurries were sampled.

SOLIDS

Coal and coke were analyzed by SSMS. No sampling methods were specified. Analytical methods were similar to those used in analysis of glass fiber filters.

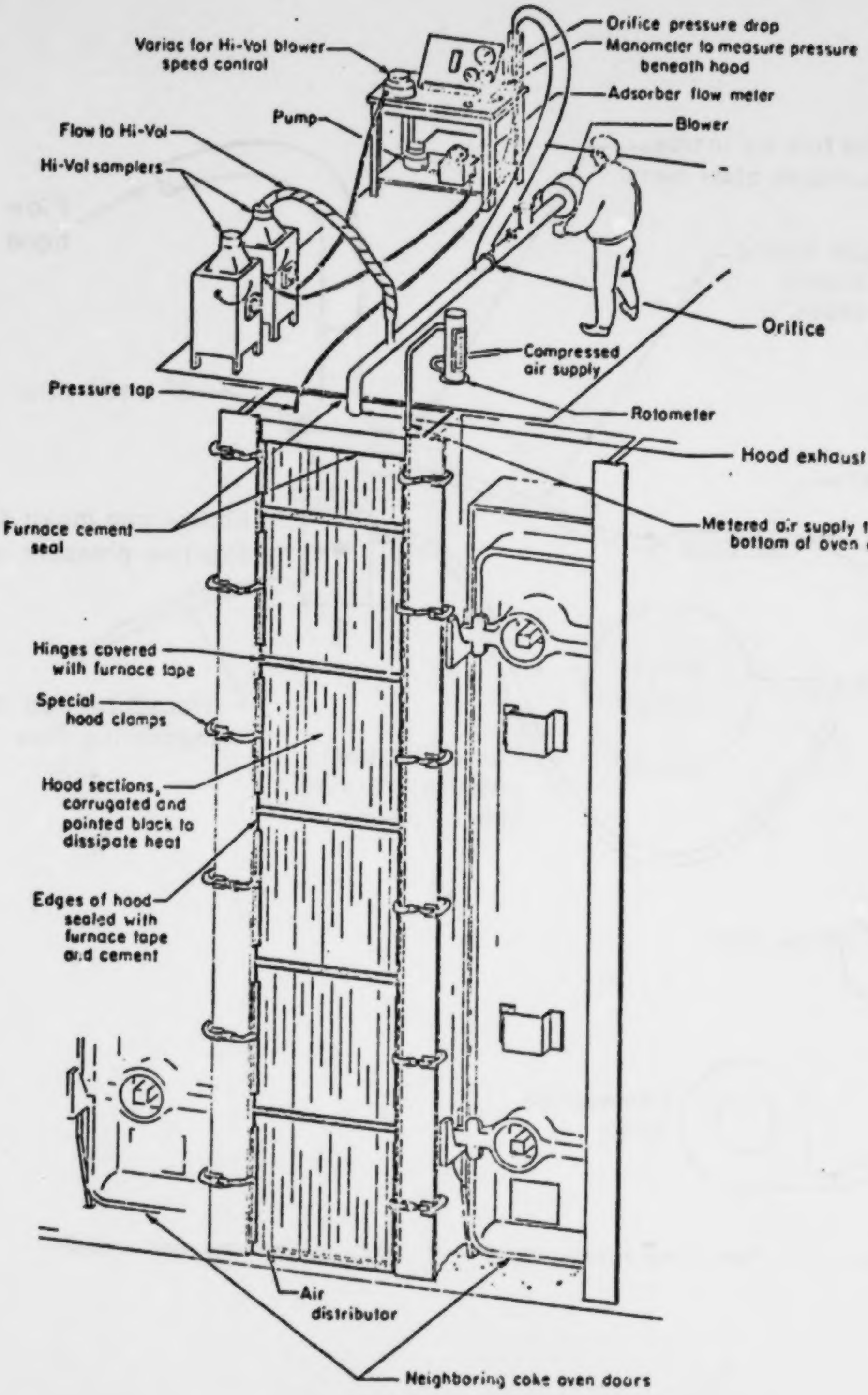
Variac for Hi-Vol blower speed control
Orifice pressure drop
Manometer to measure pressure beneath hood
Adsorber flow meter
Blower
Pump
Flow to Hi-Vol
Hi-Vol samplers
Orifice
Compressed air supply
Pressure tap
Rotometer
Hood exhaust duct
Furnace cement seal
Metered air supply to bottom of oven door
Hinges covered with furnace tape
Special hood clamps
Hood sections, corrugated and painted black to dissipate heat
Edges of hood sealed with furnace tape and cement
Air distributor
Neighboring coke oven doors

Figure 1. Test equipment arrangement on top of Oven No. 41.

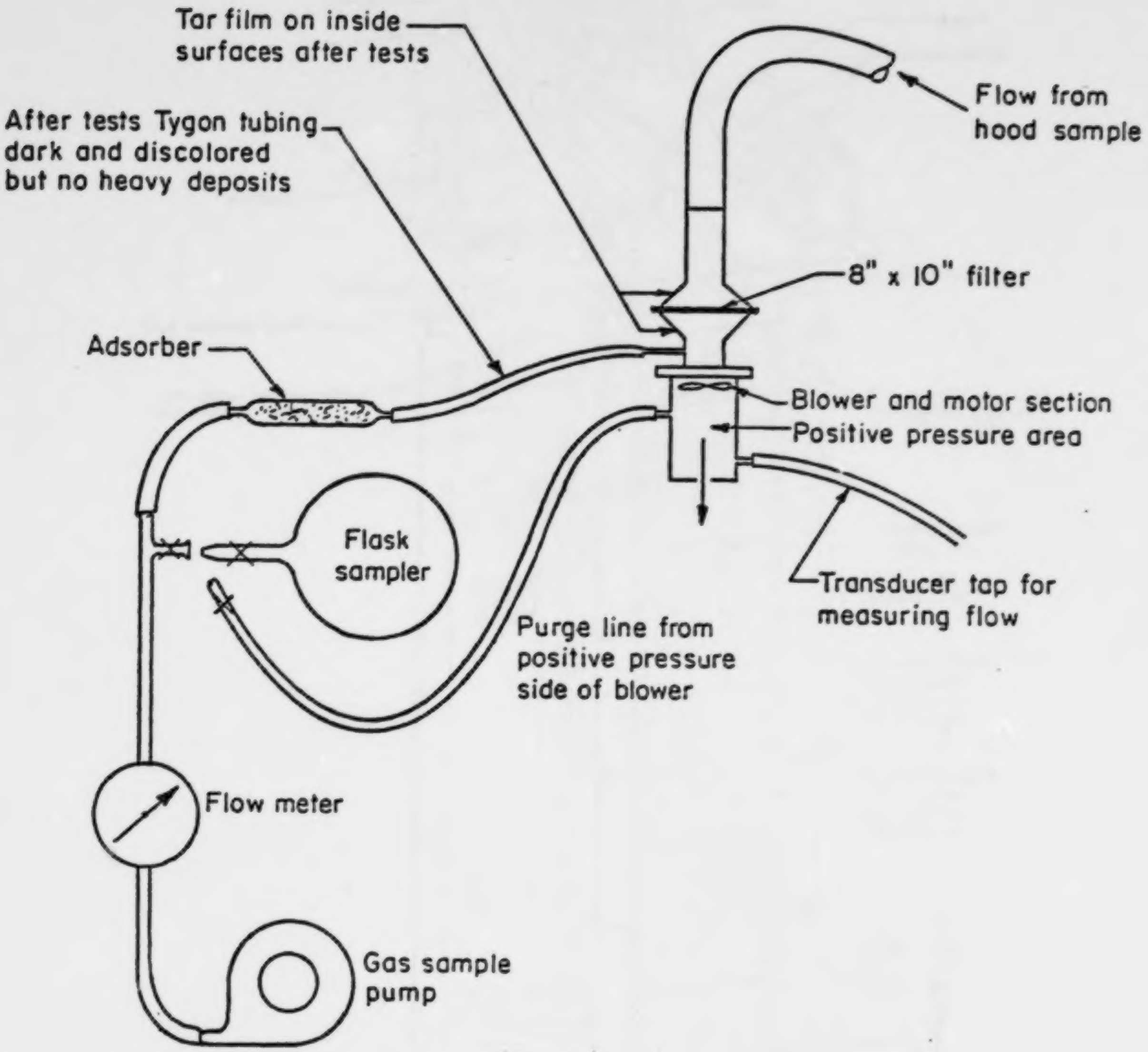
Tar film on inside surfaces after tests
After tests Tygon tubing dark and discolored but no heavy deposits
Flow from hood sample
8" x 10" filter
Adsorber
Blower and motor section
Positive pressure area
Flask sampler
Transducer tap for measuring flow
Purge line from positive pressure side of blower
Flow meter
Gas sample pump

Figure 2. Sampling arrangement for particulate and gas samples.

TABLE 1. SPARK SOURCE MASS SPECTROSCOPY
COKE OVEN DOOR EMISSIONS*--A1F
(μg/g)

U	<0.0079	Dy	<0.031	Rh	<0.031	Cr	1.6
Th	<0.0079	Tb	<0.031	Ru	<0.079	V	<0.16
Bi	0.24	Gd	<0.031	Mo	0.16	Ti	0.79
Pb	16	Eu	<0.031	Nb	<0.016	Sc	<0.31
Tl	<0.016	Sm	<0.031	Zr	0.31	Ca	<470
Hg	<0.016	Nd	<0.047	Y	<0.016	K	<310
Au	<0.016	Pr	<0.016	Sr	≤0.47	Cl	
Pt	0.016	Ce	1.6	Rb	0.47	S	<160
Ir	<0.016	La	0.79	Br	1.3	P	<0.79
Os	<0.016	Ba	<7.9	Se	<0.16	Si	
Re	<0.016	Cs	<0.016	As	0.47	Al	<110
W	0.016	I	<0.0079	Ge	<0.31	Mg	<79
Ta	<0.031	Te	<0.016	Ga	<0.16	Na	<2,400
Hf	<0.016	Sb	<0.16	Zn	≤1.6	F	<0.031
Lu	<0.031	Sn	7.9	Cu	0.47	B	<31
Yb	<0.031	In	<0.079	Ni	0.79	Be	<0.16
Tm	<0.031	Cd	<0.079	Co	0.16	Li	0.024
Er	<0.031	Ag	0.16	Fe	16		
Ho	<0.031	Pd	<0.031	Mn	0.16		

*Aqua regia extraction of Hi-Vol filter. Data expressed in μg above the blank for entire sample collected.
No data indicates "not detected above the level of the blank."

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY
COKE OVEN DOOR EMISSIONS*--A2F
(μg/g)

U	<0.016	Dy	<0.064	Rh	<0.064	Cr	1.6
Th	<0.016	Tb	<0.064	Ru	<0.16	V	<0.32
Bi	0.16	Gd	<0.064	Mo	<0.32	Ti	<1.6
Pb	<32	Eu	<0.064	Nb	<0.032	Sc	<0.64
Tl	<0.032	Sm	<0.064	Zr	<0.64	Ca	<950
Hg	<0.032	Nd	<0.095	Y	<0.032	K	<640
Au	<0.032	Pr	<0.032	Sr	<0.95	Cl	
Pt	<0.032	Ce	<0.032	Rb	<0.95	S	<320
Ir	<0.032	La	<0.032	Br	0.32	P	<1.6
Os	<0.032	Ba	<16	Se	<0.32	Si	
Re	<0.032	Cs	<0.032	As	<0.64	Al	<220
W	<0.032	I	<0.016	Ge	<0.64	Mg	<160
Ta	<0.064	Te	<0.032	Ga	<0.32	Na	<4,800
Hf	<0.032	Sb	<0.032	Zn	<3.2	F	<0.064
Lu	<0.064	Sn	<0.64	Cu	0.32	B	<64
Yb	<0.064	In	<0.16	Ni	0.95	Be	<0.32
Tm	<0.064	Cd	<0.16	Co	<0.32	Li	0.32
Er	<0.064	Ag	<0.16	Fe	1.6		
Ho	<0.064	Pd	<0.064	Mn	<0.32		

*Aqua regia extraction of Hi-Vol filter. Data expressed in μg above the blank for entire sample collected.
No data indicates "not detected above the level of the blank."

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
COKE OVEN DOOR EMISSIONS*--A3F
(μg/g)

U	<0.015	Dy	<0.058	Rh	<0.058	Cr	0.29
Th	<0.015	Tb	<0.058	Ru	<0.15	V	<0.29
Bi	<0.015	Gd	<0.058	Mo	<0.29	Ti	<1.5
Pb	<29	Eu	<0.058	Nb	<0.029	Sc	<0.58
Tl	<0.029	Sm	<0.058	Zr	<0.58	Ca	<870
Hg	<0.029	Nd	<0.087	Y	<0.029	K	<580
Au	<0.029	Pr	<0.029	Sr	<0.87	Cl	
Pt	<0.029	Ce	<0.029	Rb	<0.87	S	<290
Ir	<0.029	La	<0.029	Br	<0.29	P	<1.5
Os	<0.029	Ba	<15	Se	<0.29	Si	
Re	<0.029	Cs	<0.029	As	<0.58	Al	<204
W	<0.029	I	<0.015	Ge	≤0.58	Mg	<150
Ta	<0.058	Te	<0.029	Ga	<0.29	Na	<4,400
Hf	<0.029	Sb	<0.29	Zn	<2.9	F	<0.058
Lu	<0.058	Sn	<0.58	Cu	1.5	B	<58
Yb	<0.058	In	<0.15	Ni	<0.87	Be	<0.29
Tm	<0.058	Cd	<0.15	Co	<0.29	Li	0.015
Er	<0.058	Ag	<0.15	Fe	29		
Ho	<0.058	Pd	<0.058	Mn	<0.29		

*Aqua regia extraction of Hi-Vol filter. Data expressed in μg above the blank for entire sample collected.
No data indicates "not detected above the level of the blank."

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
COKE OVEN DOOR EMISSIONS*--A4F
(μg/g)

U	<0.026	Dy	<0.10	Rh	<0.1	Cr	<0.52
Th	<0.026	Tb	<0.10	Ru	<0.26	V	<0.52
Bi	<0.026	Gd	<0.10	Mo	<0.52	Ti	<2.6
Pb	<52	Eu	<0.10	Nb	<0.052	Sc	<1.0
Tl	<0.052	Sm	<0.10	Zr	<1.0	Ca	<1,600
Hg	<0.052	Nd	<0.16	Y	<0.052	K	<1,000
Au	<0.052	Pr	<0.052	Sr	<1.6	Cl	
Pt	<0.052	Ce	<0.052	Rb	≤1.6	S	<520
Ir	<0.052	La	<0.052	Br	<0.52	P	<2.6
Os	<0.052	Ba	<26	Se	<0.52	Si	
Re	<0.052	Cs	<0.052	As	<1.0	Al	<370
W	<0.052	I	<0.026	Ge	<1.0	Mg	<260
Ta	<0.10	Te	<0.052	Ga	<0.52	Na	<7,900
Hf	<0.052	Sb	<0.52	Zn	<5.2	F	<0.10
Lu	<0.10	Sn	<1.0	Cu	0.52	B	<100
Yb	<0.10	In	<0.26	Ni	<1.6	Be	<0.52
Tm	<0.10	Cd	<0.26	Co	<0.52	Li	<0.00010
Er	<0.10	Ag	<0.26	Fe	26		
Ho	<0.10	Pd	<0.1	Mn	<0.52		

*Aqua regia extraction of Hi-Vol filter. Data expressed in μg above the blank for entire sample collected.
No data indicates "not detected above the level of the blank."

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY
COKE OVEN DOOR EMISSIONS*--A5F
(μg/g)

U	<0.087	Dy	<0.35	Rh	<0.35	Cr	1.7
Th	<0.087	Tb	<0.35	Ru	<0.87	V	<1.7
Bi	<0.087	Gd	<0.35	Mo	<1.7	Ti	<8.7
Pb	<17	Eu	<0.35	Nb	<0.17	Sc	<3.5
Tl	<0.17	Sm	<0.35	Zr	<3.5	Ca	<5,200
Hg	<0.17	Nd	<0.52	Y	<0.17	K	<3,500
Au	<0.17	Pr	<0.17	Sr	<5.2	Cl	
Pt	<0.17	Ce	<0.17	Rb	<5.2	S	<1,700
Ir	<0.17	La	<0.17	Br	<1.7	P	<8.7
Os	<0.17	Ba	<87	Se	<1.7	Si	
Re	<0.17	Cs	<0.17	As	<3.5	Al	<1,200
W	<0.17	I	<0.087	Ge	<3.5	Mg	<870
Ta	<0.35	Te	<0.17	Ga	<1.7	Na	<26,000
Hf	<0.17	Sb	<1.7	Zn	<17	F	<0.35
Lu	<0.35	Sn	<3.5	Cu	3.5	B	<35
Yb	<0.35	In	<0.87	Ni	<5.2	Be	<350
Tm	<0.35	Cd	<0.87	Co	<1.7	Li	<0.0017
Er	<0.35	Ag	<0.87	Fe	<52		
Ho	<0.35	Pd	<0.35	Mn	<1.7		

*Aqua regia extraction of Hi-Vol filter. Data expressed in μg above the blank for entire sample collected.

No data indicates "not detected above the level of the blank."

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY
COAL
(ppm)

U	<1	Dy	3	Rh	<1	Cr	100
Th	<3	Tb	<1	Ru	<2	V	100
Bi	<1	Gd	1	Mo	10	Ti	5,000
Pb	1	Eu	<1	Nb	2	Sc	30
Tl	<2	Sm	3	Zr	200	Ca	2,000
Hg	<5	Nd	10	Y	100	K	500
Au	<2	Pr	1	Sr	2,000	Cl	100
Pt	<5	Ce	20	Rb	5	S	3,000
Ir	<3	La	10	Br	10	P	300
Os	<2	Ba	100	Se	≤5*	Si	~10%
Re	<1	Cs	1	As	10	Al	~5%
W	<1	I	<1	Ge	2	Mg	5,000
Ta	<2	Te	<1	Ga	30	Na	3,000
Hf	<3	Sb	<0.5	Zn	30	F	5
Lu	<2	Sn	1	Cu	30	B	100
Yb	<1	In	<0.5*	Ni	20	Be	5
Tm	<1	Cd	10	Co	20	Li	30
Er	<1	Ag	0.5	Fe	~5%		
Ho	<0.5	Pd	<1*	Mn	300		

*From previous memory.

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY
COKE
(ppm)

U	3	Dy	10	Rh	<1	Cr	20
Th	5	Tb	1	Ru	<5	V	50
Bi	<1	Gd	10	Mo	20	Ti	3,000
Pb	10	Eu	3	Nb	10	Sc	30
Tl	<2	Sm	10	Zr	200	Ca	~2%
Hg	<5	Nd	200	Y	100	K	2,000
Au	<2	Pr	100	Sr	3,000	Cl	200
Pt	<5	Ce	200	Rb	10	S	5,000
Ir	<3	La	300	Br	20	P	300
Os	<5	Ba	1,000	Se	<20	Si	~10%
Re	<1	Cs	20	As	20	Al	~3%
W	<2	I	5	Ge	20	Mg	2,000
Ta	<2	Te	3	Ga	20	Na	3,000
Hf	<1	Sb	2	Zn	50	F	5
Lu	0.5	Sn	100	Cu	30	B	100
Yb	5	In	<2*	Ni	100	Be	3
Tm	<1	Cd	10	Co	20	Li	10
Er	5	Ag	5	Fe	~1%		
Ho	1	Pd	<10*	Mn	3,000		

*From previous memory.

TABLE 8. LC FRACTIONATION
ABSORBER EXTRACTS*

	A1 (First adsorber)				A2 (Second adsorber)			
Fraction	TCO	Grav mg/hr	Total	Total	TCO	Grav mg/hr	Total	Total
LC 1		1,966.40				3,275.03		
LC 2		2,704.20				1,429.73		
LC 3		159.29				213.51		
LC 4		335.43				613.83		
LC 5		497.72				571.13		
LC 6		103.42				143.35		
LC 7		321.35				293.83		
LC 8		5.08				7.37		

	A3 (Third adsorber)				A4 (Fourth adsorber)			
Fraction	TCO	Grav mg/hr	Total	Total	TCO	Grav mg/hr	Total	Total
LC 1		1,000.43				1,202.53		
LC 2		1,097.24				879.45		
LC 3		81.54				10.43		
LC 4		189.34				137.74		
LC 5		127.58				271.89		
LC 6		106.22				27.09		
LC 7		96.69				75.91		
LC 8		2.60				3.66		

See footnote at end of table.

TABLE 8 (con.)

	A5 (Fifth adsorber)				A6 (Compressor air supply blank)			
Fraction	TCO	Grav mg/hr	Total	Total	TCO	Grav mg/hr	Total	Total
LC 1		581.42				20.98		
LC 2		1,067.07				3.26		
LC 3		44.56				1.01		
LC 4		106.57				0.96		
LC 5		231.11				1.10		
LC 6		17.17				0.91		
LC 7		64.89				6.37		
LC 8		2.89				0.18		

*Adsorbent samples extracted with pentane.

TABLE 9. LC FRACTIONATION FILTER EXTRACTS*

	A1F (First filter group)				A2F (Second filter group)			
Fraction	TCO	Grav g/hr	Total	Total	TCO	Grav g/hr	Total	Total
LC 1		8.28				2.79		
LC 2		22.71				20.36		
LC 3		7.93				4.95		
LC 4		6.62				3.75		
LC 5		2.88				3.74		
LC 6		0.87				0.96		
LC 7		10.09				9.25		
LC 8		0.50				0.61		

	A3F (Third filter group)				A4F (Fourth filter group)			
Fraction	TCO	Grav g/hr	Total	Total	TCO	Grav g/hr	Total	Total
LC 1		1.52				0.88		
LC 2		11.70				4.99		
LC 3		3.44				1.33		
LC 4		2.60				1.20		
LC 5		1.40				0.88		
LC 6		1.68				0.58		
LC 7		3.94				1.39		
LC 8		0.74				0.10		

*Filters extracted with methylene chloride and methanol.

TABLE 9 (con.)

	A5F (Fifth filter group)			
Fraction	TCO	Grav g/hr	Total	Total
LC 1		0.31		
LC 2		0.15		
LC 3		0.33		
LC 4		0.26		
LC 5		0.23		
LC 6		0.08		
LC 7		0.30		
LC 8		0.01		

TABLE 10. IR REPORT
SAMPLE: LC ADSORBER FRACTIONS

LC	A1	A2	A3
1	Aliphatic HC	Aliphatic HC, naphthalene, LC 1*	Aliphatic HC, napthalene
2	Aliphatic HC, fused ring aromatics†	Aliphatic HC, napthalene, fused ring aromatics~	Aliphatic HC, napthalene fused ring aromatics†
3	Nitrile‡§, fused ring aromatics† Carbazole#, LC 3*	Carbazole#, nitrile‡	Carbazole#, LC 3*
4	Conjugated ketone, nonconjugated ketone A, phenols, carbazole#, LC 4*	Conjugated ketone§, nonconjugated ketone A, phenols	Conjugated ketone§, nonconjugated ketone A, phenols
5	Conjugated ketone, nonconjugated ketone A, nonconjugated ketone B, phenols, LC 5*	Conjugated ketone§, nonconjugated ketone A, nonconjugated ketone B, phenols	Conjugated ketone§, nonconjugated ketone A, phenols.
6	Nonconjugated ketone B	Nonconjugated ketone B, LC 6*	Nonconjugated ketone B
7	Nonconjugated ketone B	Nonconjugated ketone B	Nonconjugated ketone B, LC 7*
8	Insufficient sample	Insufficient sample	Insufficient sample

*Sample subjected to GC-MS analysis.

†Pyrenes and benzo pyrenes are likely in this fraction.

‡Nitrile is most likely, but the possibility of C≡C cannot be excluded. Aromatic Para-Substitution nitrile is most likely.

§Can be conjugated ketone, quinone, or mixture of both.

#Probably primarily carbazole.

TABLE 11. IR REPORT
SAMPLE: LC ADSORBER FRACTIONS

LC	A4	A5	A6 (Compressor air supply blank)
1	Aliphatic HC, napthalene	Aliphatic HC, napthalene	Aliphatic HC
2	Aliphatic HC, napthalene, fused ring aromatics*	Aliphatic HC, napthalene, fused ring aromatics*, LC 2†	Aliphatic HC, silicone, LC 2†
3	Insufficient sample	Carbazole	Trace aromatics, LC 3†
4	Conjugated ketone‡, nonconjugated ketone A, phenols	Phenols‡, conjugated ketone‡, nonconjugated ketone A	Trace aromatics
5	Conjugated ketone‡, nonconjugated ketone A, nonconjugated ketone B, phenols	Phenols‡, nonconjugated ketone A, nonconjugated ketone B	Insufficient sample
6	Nonconjugated ketone B	Phthalate ester, nonconjugated ketone B	Insufficient sample
7	Nonconjugated ketone B	Phthalate ester, LC 7†, nonjugated ketone B	Nonconjugated ketone, phthalate, LC 7†
8	Insufficient sample	Insufficient sample	Insufficient sample

*Pyrenes and benzo pyrenes are likely in this fraction.
†Sample subjected to GC-MS analysis.
‡Can be conjugated ketone, quinone, or mixture of both.

TABLE 12: IR REPORT
SAMPLE: LC FILTER FRACTIONS

LC	A1F	A2F
1	Aliphatic HC, fused ring aromatics*	Aliphatic HC, fused ring aromatics
2	Aliphatic HC, fused ring aromatics*	Aliphatic HC, fused ring aromatics
3	Fused ring aromatics*, possible carbazole, C≡N†	Fused ring aromatics, carbazole, C≡N†
4	Phenol‡, C≡N†, LC 4§	C≡N†, phenol
5	Phenol# or amine#, C≡N†, LC 5§	Phenol
6	Phenol# or amine,#	Phenol
7	Aromatic, carboxylic acid#	Aromatic, carboxylic acid, phenol
8	Insufficient sample	Insufficient sample

*Possibly contains pyrene and/or benzo pyrenes.
†Possibly C≡N or (less likely) C≡C.
‡Possibly carbazole types.
§sample subjected to GC-MS analysis.
#Possibly S compounds.

TABLE 13. IR REPORT
SAMPLE: LC FILTER FRACTIONS

LC	A3F	A4F	A5F
1	Aliphatic HC, fused ring aromatics, LC 1*	Aliphatic HC, fused ring aromatics	Aliphatic HC, fused ring aromatics†
2	Aliphatic HC, fused ring aromatics	Aliphatic HC, fused ring aromatics	Aliphatic HC, fused ring aromatics†, LC 2*
3	C≡N‡, fused ring aromatics, carbazole, phenol§, LC 3*	C≡N‡, fused ring aromatics, carbazole#, phenol§	C≡N‡, fused ring aromatics†, ketone, phenol
4	Phenol§	Phenol§	Phenol, ketone
5	Phenol§, aromatic carboxylic acid	Phenol§, aromatic carboxylic acid	Phenol, ester phthalate
6	Aromatic carboxylic acid	Aromatic carboxylic acid	Aromatic carboxylic acid, ester phthalate, LC 6*
7	Aromatic carboxylic acid, LC 7*	Aromatic carboxylic acid	Aromatic carboxylic acid, ester phthalate
8	Insufficient sample	Insufficient sample	Insufficient sample

*Sample subjected to GC-MS analysis.
†Possibly contains pyrene ano/or benzpyrenes.
‡Possibly C≡N or (less likely) C≡C.
§Possibly S Compounds.
#Possibly carbazole types.

STUDY NUMBER 9

DATA SOURCE:	**ORGANIC ANALYSIS FOR ENVIRONMENTAL ASSESSMENT**
DATA STATUS:	Final Report, September 1977
AUTHORS: CONTRACTOR:	L. D. Johnson and R. G. Merrill Process Measurements Branch Industrial Environmental Research Laboratory U.S. Environmental Protection Agency Research Triangle Park, NC 27711

GENERAL

Organic analyses for the IERL phased approach to environmental assessments were discussed in the first part of the paper. Some examples of Level 1 data were provided from an electric arc furnace particulate sample.

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TABLE 1. LC FRACTIONATION
ELECTRIC ARC FURNACE PARTICULATE

Fraction	TCO	Grav mg	Total	Total
LC 1		7.2		
LC 2		1.5		
LC 3		2.0		
LC 4		1.9		
LC 5		1.8		
LC 6		3.3		
LC 7		1.4		
LC 8		0.1		

TABLE 2. IR REPORT
SAMPLE: FRACTION 6, ELECTRIC ARC FURNACE PARTICULATE

Wave number (cm^{-1})	Assignment
3,500	A broad band indicating hydroxyl
1,710	Aromatic or conjugated ketone
1,510	Aromatic carbon stretch
1,455; 1,460; 1,380	Carbon-carbon scissor and wag
830, 750	Substituted aromatic

Note: The IR of fraction 1 contained only hydrocarbon bands. The spectrum of fraction 3 contained bands at 2,925; 2,915; and 2,830 cm^{-1}, indicative of aliphatic substitution. Infrared analysis of fractions 3 through 7 showed that the organic content of the sample was aromatic in nature with a variety of functional groups including multiple ring structures and oxidation products such as ketones and acids. No LRMS was performed on these samples since the quantity of material in any of the fractions was less than the threshold amount.

STUDY NUMBER 10

DATA SOURCE:

COMPREHENSIVE ANALYSIS OF EMISSIONS FROM EXXON FLUIDIZED-BED COMBUSTION MINIPLANT UNIT

DATA STATUS: Draft Report, September 9, 1977

AUTHORS: J. M. Allen, J. E. Howes, Jr.
S. E. Miller, and K. M. Duke

CONTRACTOR: Battelle-Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201
Contract No. 68-02-2138

PROJECT OFFICER: D. B. Henschel
Industrial Environmental Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

Data from a Level 1 environmental assessment of Exxon's 12.5-inch (miniplant) pressurized fluidized-bed combustion (PFBC) unit located in Linden, New Jersey, are compiled in this report. Battelle Columbus Laboratories (BCL) sampled and analyzed five runs while burning Champion Pittsburgh Seam Coal (2% S) with dolomite ($CaCO_3$ + $MgCO_3$) sorbent injection. Sorbent regenerator was not used. Runs 1, 3, 4, and 5 operated at 890°-895° C; run 2 operated at 805° C.

List of procedures different from Level 1.

1. Containers for SASS samples and preconditioning cleaning procedures of containers were not as specified by Level 1. Specific variations are listed in table 1. Glass condensation modules, instead of stainless steel, were used in runs 2, 4, and 5 (acknowledged by EPA).
2. LC silica gel was activated by heating at 200° C for 24 hours prior to slurry packing the columns using pentane. Level 1 states heating at 110° C for at least 2 hours prior to dry packing the columns using methylene chloride.
3. Several inorganic gases and components were not analyzed by GC, but by various wet chemistry methods and continuous withdrawal analyzers as noted on the data sheets.
4. Samples for SSMS, except XAD-2 resin and coal, were mixed directly with graphite to form electrodes and thus bypassed the Parr bomb step. Coal was prepared by low temperature (plasma) ashing, and As, Hg, and Sb were prepared by wet digestion. Sb was analyzed by AAS with graphite furnace.

The study produced the following conclusions:

1. Except for trace elements (unresolved at time of report) "no direct emissions . . . expected to be grossly unacceptable for environmental reasons;"
2. SO_2 levels decreased during the runs, perhaps due to inadvertent use of lower sulfur coal (0.6%) as delivered by the supplier;
3. Particulate emissions were in excess of allowable emissions;
4. Organic and hydrocarbon emissions were very low;
5. Organic characterization by LC and IR resulted in many anomalies;
6. Organic analysis of coal shows "surprisingly small quantity of POM and HC compounds" with C >6;
7. Trace metals were found that exceeded land MATE values;
8. Several toxic elements in dolomite and suspended particulates exceeded land MATE values;

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9. Bed reject leachates were found to be the most toxic material in the soil microcosm test;

10. Alternate sample preparation for SSMS indicates "agreement well within Level 1 accuracy requirements" when results are compared to NBS/SRM samples.

GASEOUS GRABS

Flue gases were sampled continuously for SO_2 by IR or UV, CO by NDIR, O_2 by point electrode, and CO_2 by IR. Grab samples were taken and analyzed for O_2 by Orsat, H_2S/COS by GC, and NO_x by chemiluminescence. O_2 in dilution air was also determined continuously using a paramagnetic technique. Samples for HC analysis by FID were taken by Tedlar bags from sampling points 1a and 14 or continuously from 1p (see attached diagram).

SASS

A SASS train was used to sample flue gases. Runs 1 and 3 used a stainless steel condenser module, whereas runs 2, 4, and 5 used glass. Results moderately favor the all-glass module. Samples were collected at a flow rate of 4 scfm while located at sampling port 1a (≅ atmospheric pressure). XAD-2 sorbent was used to collect organics.

FUGITIVE EMISSIONS

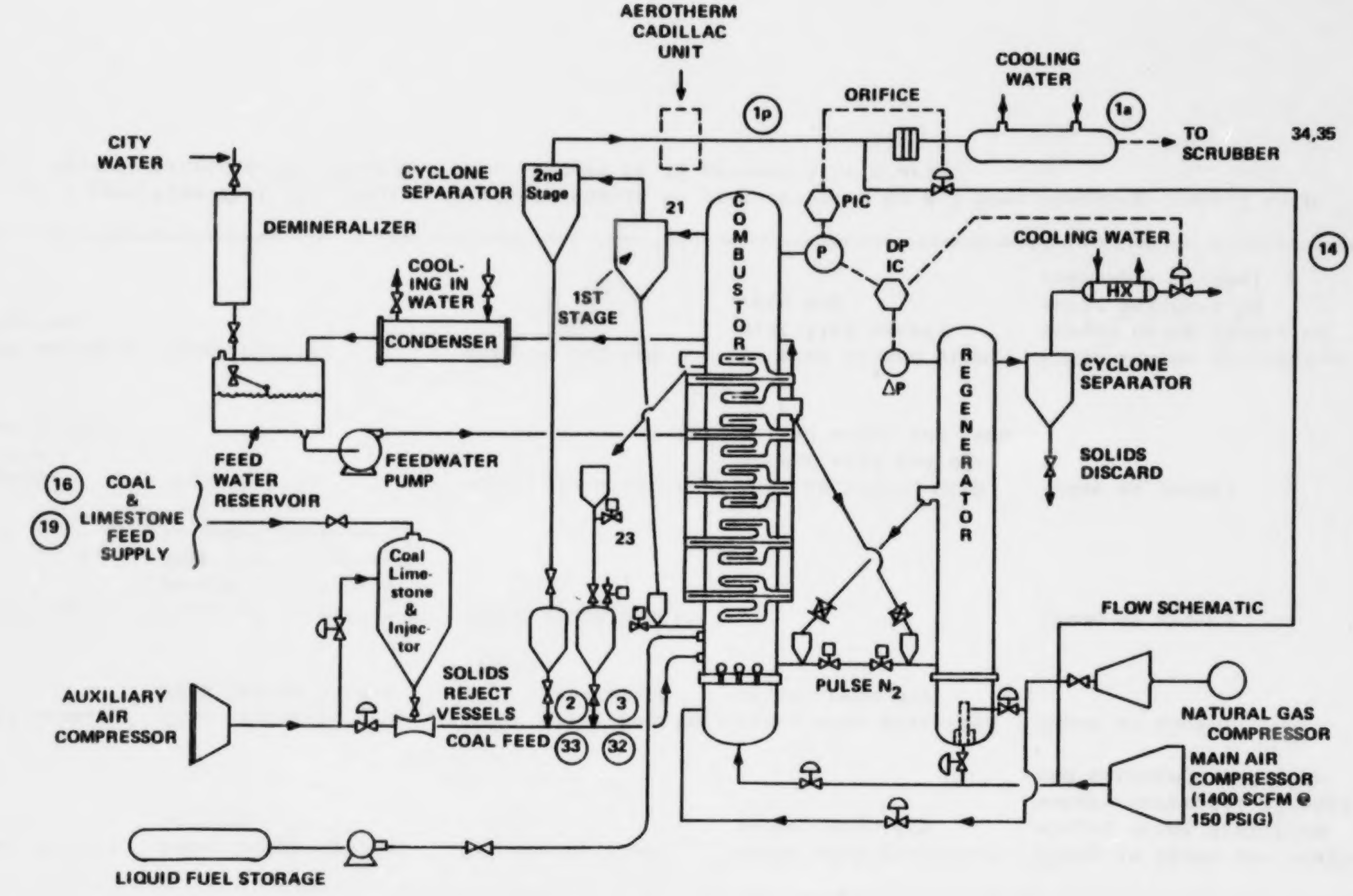
Leachates of PFBC cyclone number 2 and bed reject material were analyzed by SSMS for trace metals, toxic elements, and ions. Two leaching techniques were used. One involved ultrasonic mixing of the 1-g sample with 10 separate 4-ml volumes of distilled water. Shakes number 1 and 10 were then analyzed. The second method of leachate preparation was by pumping water through a column housing the sample.

LIQUIDS AND SLURRIES

Eight biological systems were used for wastewater toxicity evaluation. No other sampling or testing on these streams was performed.

SOLIDS

Particulate mass concentrations were determined using an HVSS with four specially designed impingers. An SS probe heated to 205° C was used at sample port 1a (alternated with SASS). Coal, dolomite, bed reject material, and PFBC cyclone Number 2 dust were all grab sampled and riffled into aliquots. Coal, PFBC cyclone Number 2 dust, and bed reject materials were Soxhlet extracted with CH_2Cl_2.


AEROTHERM
CADILLAC
UNIT
COOLING
WATER
ORIFICE
1p
1a
TO
SCRUBBER
34,35
CITY
WATER
CYCLONE
SEPARATOR
2nd
Stage
21
PIC
DP
IC
COOLING WATER
14
DEMINERALIZER
COOL-
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CYCLONE
SEPARATOR
FEED
WATER
RESERVOIR
FEEDWATER
PUMP
SOLIDS
DISCARD
16
19
COAL
&
LIMESTONE
FEED
SUPPLY
23
Coal
Lime-
stone
&
Injec-
tor
FLOW SCHEMATIC
SOLIDS
REJECT
VESSELS
2
3
PULSE N2
AUXILIARY
AIR
COMPRESSOR
COAL FEED
33
32
NATURAL GAS
COMPRESSOR
MAIN AIR
COMPRESSOR
(1400 SCFM @
150 PSIG)
LIQUID FUEL STORAGE

Circled numbers denote streams sampled, uncircled numbers were not sampled.

Figure 1. Exxon fluidized-bed combustion miniplant.

TABLE 1. CLEANING PROCEDURES FOR SAMPLE CONTAINERS

Sample	Container		Container Conditioning	
	Battelle	Level 1	Battelle	Level 1*
SASS cyclone catch	Amber polypropylene bottles	Nalgene bottles	Rinse with distilled water; oven dry	Clean in three successive stages using distilled water, isopropyl alcohol, and methylene chloride
SASS washes	Glass bottles, caps with Teflon liners	Amber glass bottles, caps with Teflon liners	Rinse with distilled water; oven dry	(Same as above)
XAD-2 resin	Original cartridge packed in polyethylene bag and stored in metal container	Amber glass bottle		(Same as above)
Organic module condensate	Glass bottle	Amber glass bottle	Bottles rinsed with nitric acid and distilled water and oven dried	(Same as above)
SASS impinger solutions	Glass bottles	Nalgene bottles	Bottles rinsed with distilled water; oven dry	Clean in two successive stages using distilled water followed by isopropyl alcohol

*Level 1 specifies that all sample containers should be prepassivated by a 1-hour standing contact with 1:1 (v/v) aqueous nitric acid. This has been changed to 15 percent nitric acid.

TABLE 2. STREAM IDENTIFICATION FOR GENERALIZED FBC SYSTEM

Stream number	Stream identification
1a	Stack gas from FBC (nominally atmospheric, after air dilution)
1p	Pressurized flue gas (before air dilution)
2	Particulate removal discard from FBC, i.e., second cyclone catch
3	Bed solids discard from FBC
14	Air to combustor (dilution air)
16	Fuel feed to FBC (coal)
19	Prepared sorbent feed to FBC (dolomite)
23	Recycle of particulates from particulate removal to FBC (first cyclone) collected only at end of test program
32	Fugitive or secondary emission from FBC discard bed material, i.e., leachate
33	Fugitive or secondary emission from FBC particulate disposal (second cyclone), i.e., leachate
35	Discard from FBC secondary stack gas cleaning device (scrubber slurry)

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY--FBC COAL RUN 2 (μg/g)

U	0.6	Dy	0.3	Rh	<0.03	Cr	24
Th	2	Tb	0.05	Ru	<0.06	V	16
Bi	<0.02	Gd	0.1	Mo	2	Ti	800
Pb	24	Eu	0.08	Nb	0.2	Sc	2
Tl	0.06	Sm	0.8	Zr	5	Ca	2,000 (0.14)*
Hg	<0.2	Nd	1	Y	0.5	K	2,000
Au	<0.02	Pr	0.5	Sr	24	Cl	2,000
Pt	<0.06	Ce	8	Rb	24	S	2,000
Ir	<0.03	La	5	Br	16	P	32
Os	<0.06	Ba	240	Se	6	Si	3.2%
Re	<0.03	Cs	0.5	As	6	Al	1%
W	0.1	I	<0.03	Ge	0.3	Mg	640
Ta	≤2	Te	<0.03	Ga	3	Na	120 (0.02)*
Hf	1	Sb	2	Zn	32	F	0.2
Lu	<0.03	Sn	0.1	Cu	16	B	5
Yb	0.2	In	<0.1	Ni	16	Be	0.1
Tm	<0.03	Cd	0.05	Co	6	Li	8
Er	0.2	Ag	0.03	Fe	6,400		
Ho	0.05	Pd	<0.03	Mn	8		

*Weight percent by AAS/flame emission.

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY--FBC COAL RUN 5 (μg/g)

U	0.6	Dy	0.3	Rh	<0.03	Cr	36
Th	2	Tb	<0.03	Ru	<0.06	V	16
Bi	<0.02	Gd	0.1	Mo	2	Ti	400
Pb	32	Eu	0.08	Nb	0.2	Sc	1
Tl	<0.06	Sm	0.8	Zr	4	Ca	1,000 (0.06)*
Hg	<0.2	Nd	1	Y	0.5	K	2,000
Au	<0.02	Pr	0.5	Sr	14	Cl	670
Pt	<0.06	Ce	8	Rb	18	S	2,000
Ir	<0.03	La	3	Br	16	P	32
Os	<0.06	Ba	120	Se	3	Si	6.4%
Re	<0.03	Cs	0.5	As	10	Al	2.4%
W	<0.1	I	<0.03	Ge	0.3	Mg	6,400
Ta	≤1	Te	<0.03	Ga	3	Na	280 (0.01)*
Hf	1	Sb	1	Zn	32	F	0.2
Lu	<0.03	Sn	0.2	Cu	16	B	5
Yb	0.2	In	<0.1	Ni	6	Be	0.1
Tm	<0.03	Cd	0.05	Co	3	Li	8
Er	0.1	Ag	0.03	Fe	6,400		
Ho	0.03	Pd	<0.03	Mn	5		

*Weight percent by AAS/flame emission.

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY--NBS/SRM 1632 COAL (μg/g)*

U	1	(1.4 NBS)	Dy	0.3		Rh	<0.03		Cr	32	(20.2 NBS)
Th	2	(3.0 NBS)	Tb	0.03		Ru	<0.06		V	16	(35 NBS)
Bi	<0.02		Gd	0.1		Mo	3	(3.4)	Ti	1,000	(800 NBS)
Pb	32	(30 NBS)	Eu	0.2	(0.21)	Nb	0.2		Sc	1	(4.5)
Tl	0.06		Sm	1	(1.7)	Zr	6	(45)	Ca	1%	(0.44%)
Hg	<0.2	(0.12 NBS)	Nd	2		Y	0.5		K	2,400	(0.29%)
Au	<0.02		Pr	0.6		Sr	160	(144)	Cl	640	(1,000)
Pt	<0.06		Ce	14	(18.5)	Rb	38	(24)	S	4,800	
Ir	<0.03		La	5	(10.5)	Br	16	(14.2)	P	24	
Os	<0.06		Ba	670	(405)	Se	6	(2.9 NBS)	Si	6.9%	(3.2% (NBS)
Re	<0.03		Cs	0.1	(1.4)	As	7	(5.9 NBS)	Al	2.7%	(1.9%)
W	<0.1		I	<0.03		Ge	0.6		Mg	3,800	(.25%)
Ta	≤2	(0.17)	Te	<0.03		Ga	5	(8.5)	Na	280	(390)
Hf	1	(0.95)	Sb	7	(4.45)	Zn	70	(37 NBS)	F	0.5	
Lu	<0.03		Sn	0.03		Cu	64	(18 NBS)	B	8	
Yb	<0.02		In	<0.1	(0.07)	Ni	14	(15 NBS)	Be	0.1	
Tm	<0.03		Cd	0.1	(0.19 NBS)	Co	13	(6 NBS)	Li	8	
Er	<0.1		Ag	0.06		Fe	2%	(0.87% NBS)			
Ho	0.03		Pd	<0.03		Mn	32	(40 NBS)			

*Values in parentheses are reference values from NBS (as noted), ORNL or LLL.

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY--NBS/SRM 88a DOLOMITE (μg/g)*

U	0.15	Dy	<0.3	Rh	<0.1		Cr	20	
Th	0.15	Tb	<0.1	Ru	<0.6		V	2	
Bi	<0.15	Gd	<0.3	Mo	0.7		Ti	50	(0.02)
Pb	0.4	Eu	0.15	Nb	0.07		Sc	<0.3	
Tl	<0.2	Sm	<0.5	Zr	0.6		Ca	30%	(30.1)
Hg	<0.4	Nd	1	Y	0.7		K	5,000	
Au	<0.1	Pr	0.5	Sr	50	(0.01)	Cl	15	
Pt	<0.4	Ce	3	Rb	6		S	500	
Ir	<0.2	La	1	Br	0.6		P	15	(0.01)
Os	<0.4	Ba	15	Se	0.3		Si	5,000	(1.2)
Re	<0.2	Cs	<0.07	As	2		Al	1,500	(0.19)
W	<0.4	I	<0.07	Ge	<0.15		Mg	15%	(21.3)
Ta	≤1	Te	<0.3	Ga	0.2		Na	30	(0.01)
Hf	≤0.3	Sb	<0.1	Zn	10		F	0.3	
Lu	<0.1	Sn	<0.2	Cu	1.5		B	1	
Yb	<0.3	In	<0.2	Ni	40		Be	0.07	
Tm	<0.1	Cd	<0.5	Co	<1		Li	3	
Er	<0.3	Ag	≤0.1	Fe	4,000	(0.28)			
Ho	<0.1	Pd	≤0.4	Mn	300	(0.03)			

*Values in parentheses are reference values from NBS (as noted), ORNL or LLL (values in percent by weight as oxides).

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY--FBC DOLOMITE* RUN 1 (μg/g)

U	0.2	Dy	<0.3	Rh	<0.1	Cr	5
Th	0.2	Tb	<0.1	Ru	<0.6	V	2
Bi	<0.15	Gd	<0.3	Mo	0.5	Ti	20
Pb	0.7	Eu	<0.15	Nb	0.07	Sc	<0.3
Tl	<0.2	Sm	<0.5	Zr	0.6	Ca	30%
Hg	<0.4	Nd	<0.5	Y	0.3	K	5,000
Au	<0.1	Pr	0.2	Sr	50	Cl	70
Pt	<0.4	Ce	0.5	Rb	3	S	100
Ir	<0.2	La	0.5	Br	3	P	3
Os	<0.4	Ba	2	Se	0.3	Si	1,500
Re	<0.2	Cs	<0.07	As	6	Al	700
W	<0.4	I	<0.07	Ge	0.2	Mg	15%
Ta	≤1	Te	<0.2	Ga	0.2	Na	500
Hf	<0.3	Sb	0.2	Zn	5	F	10
Lu	<0.1	Sn	<0.2	Cu	1.5	B	10
Yb	<0.3	In	<0.2	Ni	40	Be	0.015
Tm	<0.1	Cd	≤0.5	Co	<1	Li	5
Er	<0.3	Ag	<0.1	Fe	600		
Ho	<0.1	Pd	<0.2	Mn	50		

*Electrode formed directly--no Parr bomb.

TABLE 8. SPARK SOURCE MASS SPECTROSCOPY--FBC DOLOMITE* RUN 5 (μg/g)

U	0.4	Dy	<0.3	Rh	<0.1	Cr	5
Th	0.15	Tb	<0.1	Ru	<0.6	V	1
Bi	<0.15	Gd	<0.3	Mo	1	Ti	30
Pb	1.5	Eu	<0.15	Nb	<0.05	Sc	<0.3
Tl	<0.2	Sm	<0.5	Zr	0.3	Ca	15% (21.9)†
Hg	<0.4	Nd	<0.5	Y	0.3	K	5,000
Au	<0.1	Pr	0.1	Sr	30	Cl	400
Pt	<0.4	Ce	0.5	Rb	6	S	500
Ir	<0.2	La	0.3	Br	6	P	1
Os	<0.4	Ba	6	Se	0.6	Si	1,500
Re	<0.2	Cs	0.5	As	20	Al	600
W	<0.4	I	<0.07	Ge	0.7	Mg	20% (12.0)‡
Ta	≤3	Te	<0.2	Ga	0.2	Na	1,000
Hf	<0.3	Sb	<0.1	Zn	2	F	10
Lu	<0.1	Sn	<0.2	Cu	3	B	10
Yb	<0.3	In	<0.4	Ni	100	Be	0.01
Tm	<0.1	Cd	<0.5	Co	<1	Li	20
Er	<0.3	Ag	≤0.1	Fe	1,000		
Ho	<0.1	Pd	<0.2	Mn	100		

*Electrode formed directly--no Parr bomb.
†Weight percent by titration/calcium oxalate-potassium permanganate.
‡Weight percent by gravimetric/magnesium ammonium phosphate.

TABLE 9. SPARK SOURCE MASS SPECTROSCOPY--XAD-2 EXTRACTS* RUN 1 ($\mu g/m^3$)

U		Dy		Rh		Cr	207
Th		Tb		Ru		V	2
Bi		Gd		Mo	6	Ti	2
Pb	4	Eu		Nb	0.5	Sc	
Tl		Sm		Zr		Ca	28
Hg		Nd	0.1	Y		K	280
Au		Pr		Sr	0.3	Cl	124
Pt		Ce		Rb	0.1	S	1,388
Ir		La	0.08	Br	3	P	46
Os		Ba	3	Se		Si	1,968
Re		Cs		As	1	Al	
W	0.06	I		Ge		Mg	21
Ta		Te	0.2	Ga	0.08	Na	932
Hf		Sb	11	Zn	8	F	
Lu		Sn	0.2	Cu		B	2
Yb		In		Ni	1,864	Be	
Tm		Cd	0.7	Co	25	Li	0.04
Er		Ag	1	Fe	2,486		
Ho		Pd		Mn	41		

*Collected by SASS at (1a). Results corrected by subtraction of blank.
No data indicates "not detected above level of blank."

TABLE 10. SPARK SOURCE MASS SPECTROSCOPY--XAD-2 EXTRACTS* RUN 2 ($\mu g/m^3$)

U	0.6	Dy		Rh		Cr	47
Th		Tb		Ru		V	
Bi		Gd		Mo		Ti	
Pb		Eu		Nb		Sc	
Tl		Sm		Zr		Ca	6
Hg		Nd		Y		K	
Au		Pr		Sr	0.02	Cl	
Pt		Ce		Rb		S	93
Ir		La		Br		P	
Os		Ba	0.4	Se	3	Si	1,520
Re		Cs		As		Al	
W		I		Ge		Mg	
Ta		Te	1	Ga		Na	47
Hf		Sb	6	Zn		F	
Lu		Sn		Cu		B	
Yb		In		Ni	3,500	Be	
Tm		Cd		Co	9	Li	
Er		Ag	1	Fe	234		
Ho		Pd		Mn	94		

*Collected by SASS at (1a). Results corrected by subtraction of blank.
No data indicates "not detected above level of blank."

TABLE 11. SPARK SOURCE MASS SPECTROSCOPY--XAD-2 EXTRACTS* RUN 3 ($\mu g/m^3$)

U		Dy		Rh		Cr	310
Th		Tb		Ru		V	1
Bi		Gd		Mo	10	Ti	0.8
Pb	8	Eu	0.1	Nb		Sc	
Tl		Sm		Zr	0.6	Ca	49
Hg		Nd		Y		K	21
Au		Pr		Sr	0.2	Cl	62
Pt		Ce		Rb		S	83
Ir		La		Br	0.4	P	8
Os		Ba	2	Se	4	Si	
Re		Cs		As	5	Al	
W		I		Ge	3	Mg	
Ta		Te	0.8	Ga	0.08	Na	
Hf		Sb	28	Zn	2	F	
Lu		Sn	0.2	Cu		B	0.6
Yb		In		Ni	3,938	Be	
Tm		Cd		Co	8	Li	0.2
Er		Ag	0.3	Fe	2,486		
Ho		Pd		Mn	145		

*Collected by SASS at (1a). Results corrected by subtraction of blank.

TABLE 12. SPARK SOURCE MASS SPECTROSCOPY--XAD-2 EXTRACTS* RUN 4 ($\mu g/m^3$)

U		Dy		Rh		Cr	
Th		Tb		Ru		V	
Bi		Gd		Mo	2	Ti	0.2
Pb		Eu		Nb	0.04	Sc	
Tl		Sm		Zr	0.5	Ca	1
Hg		Nd		Y		K	
Au		Pr		Sr	0.05	Cl	
Pt		Ce		Rb		S	
Ir		La		Br		P	
Os		Ba		Se	3	Si	
Re		Cs		As		Al	
W		I		Ge		Mg	
Ta		Te	2	Ga		Na	
Hf		Sb	4	Zn		F	
Lu		Sn		Cu		B	
Yb		In		Ni		Be	
Tm		Cd		Co	2	Li	
Er		Ag		Fe			
Ho		Pd		Mn			

*Collected by SASS at (1a). Results corrected by subtraction of blank.

TABLE 13. SPARK SOURCE MASS SPECTROSCOPY--XAD-2 EXTRACTS* RUN 5 ($\mu g/m^3$)

U		Dy		Rh		Cr	25
Th		Tb		Ru		V	
Bi		Gd		Mo		Ti	
Pb		Eu		Nb	0.05	Sc	
Tl		Sm		Zr	0.3	Ca	1
Hg		Nd		Y		K	
Au		Pr		Sr		Cl	
Pt		Ce		Rb	0.02	S	
Ir		La	0.05	Br		P	2
Os		Ba		Se	2	Si	
Re		Cs		As		Al	
W		I		Ge		Mg	
Ta		Te		Ga		Na	
Hf		Sb		Zn		F	
Lu		Sn		Cu		B	
Yb		In		Ni		Be	
Tm		Cd		Co	3	Li	
Er		Ag		Fe			
Ho		Pd		Mn			

*Collected by SASS at (1a). Results corrected by subtraction of blank.

TABLE 14. SPARK SOURCE MASS SPECTROSCOPY--BLANK ($\mu g/g$)

U	0.08	Dy	<0.03	Rh	<0.02	Cr	25
Th	<0.1	Tb	<0.01	Ru	<0.03	V	0.2
Bi	<0.01	Gd	<0.03	Mo	0.5	Ti	0.2
Pb	1	Eu	<0.02	Nb	<0.004	Sc	<0.03
Tl	<0.03	Sm	<0.03	Zr	<0.05	Ca	0.8
Hg	<0.03	Nd	<0.03	Y	<0.02	K	8
Au	<0.01	Pr	<0.005	Sr	0.03	Cl	20
Pt	<0.03	Ce	0.2	Rb	0.02	S	15
Ir	<0.02	La	0.005	Br	0.1	P	2
Os	<0.03	Ba	0.08	Se	0.4	Si	25
Re	<0.02	Cs	<0.005	As	0.08	Al	75
W	0.04	I	0.08	Ge	0.08	Mg	15
Ta	<0.3	Te	0.2	Ga	0.03	Na	25
Hf	<0.03	Sb	0.8	Zn	2	F	<0.03
Lu	<0.01	Sn	0.2	Cu	250	B	0.4
Yb	<0.03	In	<0.005	Ni	300	Be	0.008
Tm	<0.01	Cd	0.08	Co	2	Li	0.02
Er	<0.03	Ag	0.05	Fe	150		
Ho	<0.01	Pd	<0.02	Mn	15		

TABLE 15. SPARK SOURCE MASS SPECTROSCOPY BASIFIED CONDENSATES FROM SASS XAD-2 RUN 5 ($\mu g/m^3$)

U	<0.0001	Dy	<0.0002	Rh	<0.0002	Cr	3
Th	<0.0001	Tb	<6 x 10^{-5}	Ru	<0.0006	V	0.0003
Bi	<0.0002	Gd	<0.0002	Mo	0.001	Ti	0.06
Pb	<0.006	Eu	<0.0002	Nb	0.002	Sc	0.0002
Tl	<0.0003	Sm	<0.0003	Zr	0.009	Ca	0.7
Hg	<0.0006	Nd	0.0006	Y	9 x 10^{-5}	K	0.06
Au	<0.0003	Pr	0.0002	Sr	0.03	Cl	0.09
Pt	<0.0003	Ce	0.0006	Rb	<3 x 10^{-5}	S	0.01
Ir	<0.0001	La	0.0006	Br	0.009	P	0.0003
Os	<0.0003	Ba	0.06	Se	0.003	Si	0.2
Re	<0.0001	Cs	<3 x 10^{-5}	As	0.003	Al	0.09
W	<0.0002	I	0.01	Ge	<0.009	Mg	0.5
Ta	≤0.001	Te	<0.0002	Ga	0.0003	Na	3
Hf	<0.0002	Sb	<0.0003	Zn	0.006	F	0.001
Lu	<6 x 10^{-5}	Sn	0.0006	Cu	0.2	B	0.003
Yb	<.0002	In	<0.0001	Ni	2	Be	<3 x 10^{-6}
Tm	<6 x 10^{-5}	Cd	<0.0009	Co	0.06	Li	6 x 10^{-5}
Er	<0.0002	Ag	<0.03	Fe	14		
Ho	<6 x 10^{-5}	Pd	<0.0001	Mn	0.2		

TABLE 16. SPARK SOURCE MASS SPECTROSCOPY BASIFIED CONDENSATES FROM SASS SECOND IMPINGER RUN 5 ($\mu g/m^3$)

U	<0.002	Dy	<0.0009	Rh	<0.0002	Cr	0.06
Th	<0.002	Tb	<0.0002	Ru	<0.0006	V	0.0009
Bi	<0.003	Gd	<0.0009	Mo	0.006	Ti	0.06
Pb	0.02	Eu	<0.003	Nb	0.0002	Sc	<9 x 10^{-5}
Tl	<0.0009	Sm	<0.0009	Zr	<0.0003	Ca	0.03
Hg	<0.003	Nd	<0.006	Y	<0.0002	K	0.03
Au	<0.0009	Pr	<0.009	Sr	0.0002	Cl	23
Pt	<0.003	Ce	<0.0002	Rb	0.0002	S	47
Ir	<0.002	La	<0.009	Br	0.06	P	0.03
Os	<0.0009	Ba	0.0009	Se	0.09	Si	2
Re	<0.0006	Cs	<0.009	As	0.002	Al	0.5
W	<0.0009	I	0.3	Ge	<0.002	Mg	0.02
Ta	≤0.02	Te	<0.0009	Ga	0.002	Na	0.09
Hf	<0.0009	Sb	0.002	Zn	≤0.02	F	<0.0009
Lu	<0.0002	Sn	0.06	Cu	0.006	B	0.006
Yb	<0.0009	In	<0.0002	Ni	0.06	Be	<6 x 10^{-5}
Tm	<0.0002	Cd	0.002	Co	0.003	Li	9 x 15^{-5}
Er	<0.0009	Ag	Major	Fe	0.9		
Ho	<0.0002	Pd	<0.0006	Mn	0.02		

TABLE 17. SPARK SOURCE MASS SPECTROSCOPY ACID CONDENSATES FROM SASS XAD-2 RUN 1 ($\mu g/m^3$)

U	0.3	Dy	<0.2	Rh	<0.2	Cr	1,519
Th	0.3	Tb	<0.1	Ru	<0.5	V	4
Bi	0.1	Gd	<0.2	Mo	30	Ti	202
Pb	2	Eu	<0.3	Nb	2	Sc	<0.1
Tl	<0.1	Sm	<0.5	Zr	3	Ca	8
Hg	<0.2	Nd	<1	Y	<0.3	K	30
Au	0.1	Pr	<0.2	Sr	2	Cl	506
Pt	0.2	Ce	<0.2	Rb	<0.3	S	10,124
Ir	<0.1	La	<0.2	Br	35	P	2
Os	<0.2	Ba	0.5	Se	<5	Si	152
Re	<0.1	Cs	0.1	As	0.5	Al	76
W	0.5	I	2	Ge	<0.2	Mg	20
Ta	≤0.6	Te	≤0.3	Ga	<0.5	Na	51
Hf	<0.2	Sb	0.5	Zn	20	F	0.1
Lu	<0.6	Sn	3	Cu	4	B	101
Yb	<0.2	In	0.06	Ni	1,519	Be	0.009
Tm	<0.6	Cd	3	Co	20	Li	0.5
Er	<0.2	Ag	506	Fe	10,124		
Ho	<0.2	Pd	<0.1	Mn	1,012		

TABLE 18. SPARK SOURCE MASS SPECTROSCOPY ACID CONDENSATES FROM SASS XAD-2 RUN 2 ($\mu g/m^3$)

U	<0.8	Dy	<0.5	Rh	<0.5	Cr	38
Th	<0.8	Tb	<0.1	Ru	<8	V	0.4
Bi	<0.1	Gd	<0.5	Mo	<0.8	Ti	88
Pb	<0.3	Eu	<0.3	Nb	<0.8	Sc	<0.3
Tl	<0.3	Sm	<0.5	Zr	<0.5	Ca	13
Hg	0.4	Nd	<0.5	Y	<0.8	K	2
Au	<0.1	Pr	<0.3	Sr	0.2	Cl	251
Pt	<0.3	Ce	<0.3	Rb	<0.1	S	5,011
Ir	<0.3	La	<0.3	Br	19	P	1
Os	<0.5	Ba	<0.1	Se	≤8	Si	1,253
Re	<0.3	Cs	<0.3	As	0.3	Al	25
W	<0.3	I	1	Ge	<0.5	Mg	251
Ta	<0.8	Te	<0.3	Ga	<1	Na	501
Hf	<0.5	Sb	0.1	Zn	9	F	<0.5
Lu	<0.1	Sn	<0.3	Cu	2	B	<0.5
Yb	<0.5	In	<0.1	Ni	88	Be	<0.006
Tm	<0.1	Cd	<0.3	Co	0.4	Li	0.5
Er	<0.5	Ag	1	Fe	251		
Ho	<0.1	Pd	<0.3	Mn	8		

TABLE 19. SPARK SOURCE MASS SPECTROSCOPY ACID CONDENSATES FROM SASS XAD-2 RUN 3 (μg/m³)

U	<0.5	Dy	<0.3	Rh	<0.3	Cr	4,918
Th	<0.5	Tb	<0.08	Ru	<5	V	6
Bi	<0.08	Gd	<0.3	Mo	164	Ti	123
Pb	0.2	Eu	<0.5	Nb	2	Sc	<0.2
Tl	<0.2	Sm	<0.3	Zr	5	Ca	49
Hg	0.5	Nd	<0.5	Y	<0.5	K	12
Au	<0.08	Pr	<0.2	Sr	1	Cl	8,197
Pt	<0.2	Ce	<0.3	Rb	5	S	1,639
Ir	<0.2	La	<0.3	Br	123	P	1
Os	<0.3	Ba	0.8	Se	≤2	Si	492
Re	<0.2	Cs	<0.2	As	0.8	Al	164
W	0.8	I	8	Ge	0.8	Mg	164
Ta	<0.3	Te	2	Ga	<0.8	Na	264
Hf	<0.3	Sb	0.6	Zn	3	F	16
Lu	<0.08	Sn	0.8	Cu	5	B	25
Yb	<0.3	In	0.2	Ni	4,918	Be	<0.003
Tm	<0.08	Cd	0.8	Co	33	Li	0.3
Er	<0.3	Ag	8	Fe	16,393		
Ho	<0.08	Pd	<0.5	Mn	82		

TABLE 20. SPARK SOURCE MASS SPECTROSCOPY ACID CONDENSATES FROM SASS XAD-2 RUN 4 (μg/m³)

U	<0.3	Dy	<0.2	Rh	<0.2	Cr	56
Th	<0.3	Tb	<0.06	Ru	<3	V	2
Bi	<0.06	Gd	<0.2	Mo	1	Ti	≤6
Pb	0.8	Eu	<0.2	Nb	2	Sc	<0.6
Tl	<0.2	Sm	<0.3	Zr	0.6	Ca	83
Hg	<0.2	Nd	<0.3	Y	0.2	K	33
Au	<0.06	Pr	<0.06	Sr	2	Cl	1,110
Pt	<0.2	Ce	0.4	Rb	<0.2	S	233
Ir	<0.1	La	0.2	Br	167	P	8
Os	<0.2	Ba	2	Se	8	Si	1,666
Re	<0.1	Cs	<0.03	As	2	Al	833
W	<0.2	I	3	Ge	0.6	Mg	833
Ta	<0.3	Te	<0.1	Ga	0.3	Na	333
Hf	<0.2	Sb	<0.1	Zn	4	F	56
Lu	<0.06	Sn	<0.6	Cu	33	B	111
Yb	<0.3	In	0.06	Ni	222	Be	0.02
Tm	<0.06	Cd	0.2	Co	56	Li	1
Er	<0.2	Ag	56	Fe	2,777		
Ho	<0.06	Pd	<0.3	Mn	56		

TABLE 21. SPARK SOURCE MASS SPECTROSCOPY ACID CONDENSATES FROM SASS XAD-2 RUN 5 ($\mu g/m^3$)

U	<0.5	Dy	0.3	Rh	<0.3	Cr	87
Th	<0.5	Tb	<0.1	Ru	<0.9	V	3
Bi	<0.1	Gd	<0.9	Mo	13	Ti	≤17
Pb	0.9	Eu	<0.3	Nb	1	Sc	<0.9
Tl	<0.3	Sm	<0.5	Zr	0.9	Ca	869
Hg	0.5	Nd	<0.5	Y	≤0.2	K	26
Au	<0.1	Pr	<0.2	Sr	1	Cl	1,739
Pt	<0.3	Ce	1	Rb	<2	S	13,039
Ir	<0.2	La	0.6	Br	522	P	1
Os	<0.3	Ba	5	Se	35	Si	2,608
Re	<0.2	Cs	0.1	As	3	Al	348
W	0.3	I	5	Ge	2	Mg	522
Ta	<0.5	Te	≤0.9	Ga	0.3	Na	174
Hf	<0.3	Sb	0.3	Zn	13	F	26
Lu	<0.3	Sn	1	Cu	5	B	87
Yb	<0.5	In	0.2	Ni	61	Be	<0.06
Tm	<0.1	Cd	0.6	Co	52	Li	0.2
Er	<0.3	Ag	52	Fe	4,346		
Ho	<0.1	Pd	<0.5	Mn	261		

TABLE 22. SPARK SOURCE MASS SPECTROSCOPY PARTICULATES OF FLUE GAS*--RUN 2 (μg/g)

	<3μ	>3μ		<3μ	>3μ
U	7	4	Pd	<2	<2
Th	20	7	Rh	<0.2	<0.2
Bi	1.5	0.7	Ru	<1	<1
Pb	100	60	Mo	10	10
Tl	7	2	Nb	20	10
Hg	<0.4	<0.4	Zr	100	100
Au	<0.1	<0.1	Y	30	30
Pt	<0.4	<0.4	Sr	700	700
Ir	<0.2	<0.2	Rb	300	300
Os	<0.4	<0.4	Br	7	7
Re	<0.2	<0.2	Se	10	10
W	2	<0.5	As	100	50
Ta	<1	<2	Ge	20	10
Hf	6	3	Ga	70	70
Lu	0.4	<0.2	Zn	200	200
Yb	6	3	Cu	70	70
Tm	1	0.5	Ni	400	200
Er	3	3	Co	30	30
Ho	1	1	Fe	6%	10%
Dy	5	5	Mn	200	200
Tb	1	1	Cr	300	300
Gd	6	3	V	200	200
Eu	3	1.5	Ti	4,000	7,000
Sm	7	4	Sc	50	70%
Nd	40	20	Ca	1.5%	1.5%
Pr	10	15	K	3%	2%
Ce	70	50	Cl	4	7
La	30	30	S	1.5%	7,000
Ba	1,000	700	P	700	500
Cs	20	10	Si	10%	20%
I	0.6	0.4	Al	10%	20%
Te	<0.3	<0.3	Mg	1%	2%
Sb	3	1	Na	1,000	2,000
Sn	10	3	F	5	10
In	<1	<0.6	B	100	100
Cd	10	10	Be	6	10
Ag	15	7	Li	300	300

*Collected by SASS at 1a. Electrode formed directly (no Parr bomb).

TABLE 23. SPARK SOURCE MASS SPECTROSCOPY
PARTICULATES OF FLUE GAS*--RUN 4
(μg/g)

	<3μ	>3μ		<3μ	>3μ
U	7	2	Pd	<2	<2
Th	15	7	Rh	<0.2	<0.2
Bi	0.4	0.7	Ru	<1	<1
Pb	100	60	Mo	5	10
Tl	4	4	Nb	5	10
Hg	<0.4	<0.4	Zr	100	300
Au	<0.1	<0.1	Y	30	50
Pt	<0.4	<0.4	Sr	700	1,500
Ir	<0.2	<0.2	Rb	600	300
Os	<0.4	<0.4	Br	4	7
Re	<0.2	<0.2	Se	10	10
W	1	1	As	40	50
Ta	<2	<2	Ge	10	10
Hf	3	3	Ga	70	100
Lu	0.4	0.4	Zn	100	100
Yb	6	3	Cu	40	70
Tm	0.3	0.5	Ni	200	400
Er	3	3	Co	30	30
Ho	1	1	Fe	3%	6%
Dy	5	7	Mn	200	200
Tb	1	1	Cr	200	300
Gd	6	6	V	200	100
Eu	3	3	Ti	4,000	7,000
Sm	7	7	Sc	20	50
Nd	40	40	Ca	3%	5%
Pr	10	15	K	1.5%	5%
Ce	70	70	Cl	4	7
La	30	30	S	4,000	2%
Ba	2,000	1,000	P	300	500
Cs	40	20	Si	10%	15%
I	0.4	<0.1	Al	10%	20%
Te	<0.3	<0.3	Mg	2%	2%
Sb	1	1	Na	2,000	2,000
Sn	3	3	F	15	15
In	<0.6	<1	B	100	100
Cd	<3	<6	Be	4	4
Ag	1	1	Li	500	300

*Collected by SASS at 1a. Electrode formed directly (no Parr bomb).

TABLE 24. SPARK SOURCE MASS SPECTROSCOPY PARTICULATES OF FLUE GAS*--RUN 5
(μg/g)

	<3μ	>3μ		<3μ	>3μ
U	7	2	Pd	<0.7	<0.4
Th	15	7	Rh	<0.4	<0.4
Bi	0.6	0.3	Ru	<1	<0.4
Pb	40	40	Mo	5	3
Tl	3	1.5	Nb	7	3
Hg	<0.4	<0.4	Zr	100	100
Au	<0.1	<0.1	Y	40	15
Pt	<0.4	<0.4	Sr	1,500	300
Ir	<0.2	<0.2	Rb	100	40
Os	<0.4	<0.4	Br	10	2.5
Re	<0.2	<0.2	Se	15	5
W	0.5	0.5	As	30	10
Ta	≤1	≤0.6	Ge	15	3
Hf	3	1	Ga	60	10
Lu	0.3	<0.15	Zn	70	20
Yb	3	2	Cu	70	30
Tm	0.6	0.15	Ni	400	100
Er	3	2	Co	30	20
Ho	1	0.6	Fe	6%	2%
Dy	4	3	Mn	200	50
Tb	0.7	0.5	Cr	300	200
Gd	5	2	V	200	50
Eu	3	1	Ti	7,000	3,000
Sm	7	3	Sc	40	70
Nd	40	10	Ca	3%	3%
Pr	7	2	K	3,000	1,500
Ce	150	50	Cl	20	20
La	50	20	S	1.5%	7,000
Ba	1,500	600	P	100	500
Cs	20	3	Si	20%	20%
I	0.4	0.1	Al	20%	20%
Te	<0.3	<0.3	Mg	10%	5%
Sb	2	0.7	Na	1,500	700
Sn	6	2	F	15	15
In	<0.6	<0.6	B	50	50
Cd	<1.5	<1.5	Be	4	3
Ag	0.7	0.7	Li	200	100

*Collected by SASS at 1a. Electrode formed directly (no Parr bomb).

TABLE 25. SPARK SOURCE MASS SPECTROSCOPY BED REJECT MATERIAL*--RUN 2
(µg/g)

U	0.4	Dy	0.6	Rh	<0.2	Cr	20
Th	1	Tb	0.3	Ru	<0.5	V	20
Bi	0.1	Gd	0.6	Mo	4	Ti	200
Pb	3	Eu	0.5	Nb	0.7	Sc	3
Tl	1	Sm	1.5	Zr	10	Ca	15%
Hg	<0.3	Nd	4	Y	3	K	1%
Au	<0.1	Pr	1	Sr	150	Cl	40
Pt	<0.3	Ce	5	Rb	40	S	3%
Ir	<0.2	La	3	Br	1	P	200
Os	<0.3	Ba	60	Se	1.5	Si	3%
Re	<0.2	Cs	4	As	30	Al	2%
W	<0.3	I	<0.07	Ge	10	Mg	15%
Ta	≤0.3	Te	<0.3	Ga	7	Na	100
Hf	‾0.3	Sb	0.7	Zn	100	F	0.6
Lu	<0.1	Sn	2	Cu	15	B	50
Yb	1	In	<0.3	Ni	100	Be	0.5
Tm	<0.15	Cd	<0.5	Co	3	Li	50
Er	0.3	Ag	≤0.1	Fe	1%		
Ho	<0.15	Pd	≤1	Mn	300		

*Electrode formed directly (no Parr bomb).

TABLE 26. SPARK SOURCE MASS SPECTROSCOPY BED REJECT MATERIAL*--RUN 4
(µg/g)

U	0.2	Dy	0.6	Rh	<0.4	Cr	30
Th	1	Tb	0.5	Ru	<1	V	30
Bi	<0.1	Gd	1	Mo	4	Ti	400
Pb	1	Eu	0.5	Nb	1.5	Sc	3
Tl	2	Sm	1.5	Zr	20	Ca	15%
Hg	<0.3	Nd	4	Y	5	K	2%
Au	<0.1	Pr	0.5	Sr	300	Cl	40
Pt	<0.3	Ce	20	Rb	40	S	1.5%
Ir	<0.2	La	6	Br	1	P	100
Os	<0.3	Ba	100	Se	1.5	Si	7%
Re	<0.2	Cs	2	As	15	Al	5%
W	<0.3	I	<0.07	Ge	7	Mg	10%
Ta	<1	Te	<0.3	Ga	15	Na	50
Hf	≤0.3	Sb	0.4	Zn	5	F	0.3
Lu	<0.1	Sn	1	Cu	15	B	100
Yb	<0.3	In	<0.6	Ni	150	Be	1
Tm	<0.1	Cd	<0.5	Co	2	Li	100
Er	<0.3	Ag	<0.1	Fe	3%		
Ho	<0.1	Pd	≤1	Mn	100		

*Electrode formed directly (no Parr bomb).

TABLE 27. SPARK SOURCE MASS SPECTROSCOPY
BED REJECT MATERIAL*--RUN 5
(μg/g)

U	0.4	Dy	0.3	Rh	<1	Cr	30
Th	0.2	Tb	1	Ru	<1	V	20
Bi	<0.15	Gd	3	Mo	2	Ti	200
Pb	2	Eu	0.5	Nb	0.5	Sc	3
Tl	<0.2	Sm	1.5	Zr	10	Ca	20%
Hg	<0.4	Nd	6	Y	1.5	K	1%
Au	<0.1	Pr	1	Sr	300	Cl	40
Pt	<0.4	Ce	7	Rb	60	S	1.5%
Ir	<0.2	La	7	Br	3	P	50
Os	<0.4	Ba	30	Se	3	Si	1.5%
Re	<0.4	Cs	4	As	20	Al	1.5%
W	<0.4	I	<0.07	Ge	40	Mg	20%
Ta	<0.3	Te	<0.4	Ga	7	Na	300
Hf	≤0.3	Sb	1	Zn	400	F	0.3
Lu	≤0.3	Sn	2	Cu	15	B	30
Yb	≤0.3	In	<0.6	Ni	150	Be	0.3
Tm	<0.1	Cd	<0.5	Co	1.5	Li	200
Er	<0.3	Ag	<0.1	Fe	1%		
Ho	<0.1	Pd	≤0.5	Mn	100		

*Electrode formed directly (no Parr bomb).

TABLE 28. SPARK SOURCE MASS SPECTROSCOPY
PFBC CYCLONE NUMBER 2 DUST*--RUN 2
(μg/g)

U	1	Dy	2	Rh	<0.4	Cr	100
Th	4	Tb	0.3	Ru	<1	V	100
Bi	<0.1	Gd	2	Mo	3	Ti	5,000
Pb	7	Eu	1	Nb	3	Sc	15
Tl	1.5	Sm	3	Zr	60	Ca	3%
Hg	<0.4	Nd	10	Y	10	K	1%
Au	<0.1	Pr	5	Sr	500	Cl	20
Pt	<0.4	Ce	50	Rb	150	S	7,000
Ir	<0.2	La	15	Br	2.5	P	500
Os	<0.4	Ba	400	Se	3	Si	15%
Re	<0.2	Cs	7	As	20	Al	15%
W	<0.3	I	<0.1	Ge	6	Mg	5%
Ta	<0.6	Te	<0.3	Ga	20	Na	3,000
Hf	<1	Sb	0.7	Zn	60	F	3
Lu	<0.1	Sn	1	Cu	30	B	50
Yb	1	In	<0.6	Ni	400	Be	3
Tm	<0.15	Cd	<1.5	Co	20	Li	200
Er	1	Ag	<0.3	Fe	10%		
Ho	0.15	Pd	<0.7	Mn	100		

*Electrode formed directly (no Parr bomb).

TABLE 29. SPARK SOURCE MASS SPECTROSCOPY PFBC CYCLONE NUMBER 2 DUST*--RUN 4 (μg/g)

U	2	Dy	4	Rh	<0.4	Cr	50
Th	4	Tb	0.7	Ru	<1	V	100
Bi	0.3	Gd	5	Mo	3	Ti	5,000
Pb	20	Eu	2	Nb	5	Sc	15
Tl	1.5	Sm	7	Zr	100	Ca	5%
Hg	<0.4	Nd	40	Y	15	K	3,000
Au	<0.1	Pr	7	Sr	700	Cl	10
Pt	<0.4	Ce	100	Rb	150	S	7,000
Ir	<0.2	La	50	Br	5	P	300
Os	<0.4	Ba	600	Se	1.5	Si	3%
Re	<0.2	Cs	10	As	10	Al	6%
W	<0.5	I	0.1	Ge	6	Mg	3%
Ta	≤1	Te	0.6	Ga	20	Na	1,500
Hf	≤2	Sb	0.7	Zn	40	F	6
Lu	0.3	Sn	3	Cu	50	B	30
Yb	3	In	<0.6	Ni	100	Be	0.15
Tm	0.3	Cd	1.5	Co	10	Li	100
Er	2	Ag	≤0.2	Fe	10%		
Ho	0.6	Pd	≤2	Mn	100		

*Electrode formed directly (no Parr bomb).

TABLE 30. SPARK SOURCE MASS SPECTROSCOPY PFBC CYCLONE NUMBER 2 DUST*--RUN 5 (μg/g)

U	2	Dy	3	Rh	<0.4	Cr	200
Th	2	Tb	1	Ru	<0.5	V	200
Bi	0.1	Gd	3	Mo	7	Ti	7,000
Pb	20	Eu	5	Nb	7	Sc	10
Tl	0.5	Sm	15	Zr	200	Ca	5%
Hg	<0.4	Nd	20	Y	50	K	6,000
Au	<0.1	Pr	10	Sr	1,500	Cl	40
Pt	<0.3	Ce	100	Rb	400	S	1.5%
Ir	<0.2	La	50	Br	1.5	P	300
Os	<0.3	Ba	2,000	Se	1.5	Si	13%
Re	<0.2	Cs	60	As	40	Al	13%
W	<0.3	I	<0.06	Ge	20	Mg	3%
Ta	<0.6	Te	0.3	Ga	70	Na	1,000
Hf	≤1	Sb	0.4	Zn	40	F	3
Lu	0.6	Sn	1	Cu	50	B	30
Yb	3	In	<0.2	Ni	300	Be	0.15
Tm	0.6	Cd	<1.5	Co	3	Li	50
Er	3	Ag	<0.2	Fe	10%		
Ho	1	Pd	<1	Mn	300		

*Electrode formed directly (no Parr bomb).

TABLE 31. SPARK SOURCE MASS SPECTROSCOPY
NBS/SRM COAL FLYASH
(μg/g)*

U	8	(11.6 NBS)	Pd	<2	
Th	15	(24 NBS)	Rh	<0.2	
Bi	0.15		Ru	<0.5	
Pb	40	(70 NBS)	Mo	7	
Tl	1.5		Nb	7	
Hg	<0.4	(0.14 NBS)	Zr	200	(301)
Au	<0.1		Y	20	
Pt	<0.3		Sr	700	(1,380 NBS)
Ir	<0.2		Rb	200	(112 NBS)
Os	<0.3		Br	15	(6.0)
Re	<0.2		Se	5	(9.4 NBS)
W	1		As	40	(61 NBS)
Ta	≤0.6	(1.6)	Ge	10	
Hf	6	(10.8)	Ga	40	(49)
Lu	0.2		Zn	200	(210 NBS)
Yb	0.2		Cu	150	(128 NBS)
Tm	0.2		Ni	100	(98 NBS)
Er	2		Co	30	(38 NBS)
Ho	0.5		Fe	10%	(6.37%)
Dy	3		Mn	500	(493 NBS)
Tb	0.5		Cr	100	(131 NBS)
Gd	3		V	200	(214 NBS)
Eu	3	(2.86)	Ti	6,000	(6,920)
Sm	5	(15)	Sc	30	(32)
Nd	20		Ca	3%	(4.34%)
Pr	3		K	1.5%	(1.72%)
Ce	100	(146)	Cl	40	(42)
La	40	(82)	S	700	
Ba	2,000	(2,780)	P	300	
Cs	20	(8.6)	Si	15%	(21%)
I	0.4		Al	15%	(12.5%)
Te	<0.2		Mg	2%	(1.98 NBS)
Sb	2	(1.8)	Na	1,000	(3,070 NBS)
Sn	0.5		F	6	
In	<0.6		B	200	
Cd	5	(1.45 NBS)	Be	3	
Ag	0.6		Li	100	

*Values in parentheses are reference values from NBS (as noted), ORNL, and LLL.

TABLE 32. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF BED REJECT MATERIAL RUN 2 (ULTRASONIC SHAKING) (μg/l)

	Shake Number			Shake Number	
	1	10		1	10
U	<2	<2	Pd	<1	<1
Th	<2	<2	Rh	<1	<2
Bi	<0.4	<0.5	Ru	<2.5	<3
Pb	1.5	3	Mo	25	10
Tl	<0.7	<1	Nb	<0.6	<0.7
Hg	<2	<2	Zr	2.5	5
Au	<2	<2	Y	6	<0.7
Pt	<2	<2	Sr	600	700
Ir	<0.7	<1	Rb	4	100
Os	<2	<2	Br	3	3
Re	<0.7	<1	Se	40	50
W	<2	<2	As	15	30
Ta	≤60	≤70	Ge	<1.5	<2
Hf	<1	<2	Ga	2	1
Lu	<0.4	<0.5	Zn	<40	<50
Yb	<1	<2	Cu	20	20
Tm	<0.4	<0.5	Ni	20	70
Er	<1	<2	Co	<4	5
Ho	<0.4	<0.5	Fe	250	500
Dy	<1	<2	Mn	4	10
Tb	<0.4	<0.5	Cr	40	50
Gd	<1	<2	V	7	10
Eu	<0.7	<2	Ti	70	<50
Sm	<1	<3	Sc	<1.5	<1
Nd	<1	<2	Ca	300,000	400,000
Pr	<0.4	<0.5	K	4,000	7,000
Ce	0.4	0.7	Cl	70	300
La	0.6	0.5	S	150,000	250,000
Ba	70	300	P	100	150
Cs	2	1	Si	2,000	1,000
I	0.4	0.5	Al	600	500
Te	<1	<2	Mg	20,000	1,500
Sb	<0.3	<1	Na	7,000	1,500
Sn	2.5	3	F	<1.5	<2
In	<0.4	<0.5	B	200	30
Cd	<1.5	<2	Be	0.03	<0.07
Ag	<1	<1	Li	400	200

*See description of leachate preparation in summary. Electrode formed directly (no Parr bomb).

TABLE 33. SPARK SOURCE MASS SPECTROSCOPY
LEACHATE* OF BED REJECT MATERIAL
RUN 2 (COLUMN)
(μg/l)

U	<15	Tb	<3	Ru	<30	V	600
Th	<15	Gd	<10	Mo	100	Ti	1,500
Bi	<3	Eu	<15	Nb	<2	Sc	<10
Pb	30	Sm	<15	Zr	20	Ca	2,000,000
Tl	<6	Nd	<10	Y	<10	K	20,000
Hg	<15	Pr	<3	Sr	3,000	Cl	5,000
Au	<15	Ce	2	Rb	2,000	S	5,000,000
Pt	<15	La	20	Br	200	P	1,000
Ir	<6	Ba	1,500	Se	500	Si	3,000
Os	<15	Cs	200	As	300	Al	500
Re	<6	I	3	Ge	<15	Mg	3,000,000
W	20	Te	<10	Ga	15	Na	50,000
Ta	≤30	Sb	15	Zn	<300	F	<10
Hf	<10	Sn	10	Cu	100	B	600
Lu	<3	In	<6	Ni	500	Be	0.2
Yb	<10	Cd	<20	Co	<30	Li	3,000
Tm	<3	Ag	3	Fe	4,000		
Er	<10	Pd	<6	Mn	100		
Ho	<3	Rh	<15	Cr	300		
Dy	<10						

*Electrode formed directly (no Parr bomb).

TABLE 34. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF BED REJECT MATERIAL RUN 4 (ULTRASONIC SHAKING) (μg/1)

	Shake Number			Shake Number	
	1	10		1	10
U	<3	<2	Pd	<2	<2
Th	<3	<2	Rh	<3	<1
Bi	<0.6	<0.3	Ru	<10	<3
Pb	10	2	Mo	100	6
Tl	<1	<0.6	Nb	0.3	<0.5
Hg	<3	<2	Zr	1	6
Au	<3	<2	Y	<0.3	0.2
Pt	<3	<2	Sr	400	500
Ir	<1	<0.6	Rb	60	30
Os	<3	<2	Br	15	2
Re	<1	<0.6	Se	30	30
W	<2	<1	As	30	10
Ta	≤60	≤30	Ge	<3	<1
Hf	<2	<1	Ga	1	2
Lu	<0.6	<0.3	Zn	<60	<30
Yb	<2	<1	Cu	150	15
Tm	<0.6	<0.3	Ni	150	20
Er	<2	<1	Co	<6	<3
Ho	<0.6	<0.3	Fe	3,000	300
Dy	<2	<1	Mn	30	10
Tb	<1	<0.3	Cr	100	15
Gd	<2	<1	V	30	6
Eu	<3	<1	Ti	600	60
Sm	<3	<2	Sc	<2	<1
Nd	4	<1	Ca	600,000	300,000
Pr	<0.4	<0.3	K	15,000	3,000
Ce	1	0.5	Cl	400	60
La	1.5	0.3	S	400,000	200,000
Ba	400	200	P	3,000	30
Cs	6	2	Si	3,000	600
I	1	0.6	Al	3,000	300
Te	6	<1	Mg	10,000	3,000
Sb	1	<0.3	Na	15,000	2,000
Sn	6	1	F	6	3
In	<1	<0.3	B	10	30
Cd	<3	<1	Be	<0.03	<0.05
Ag	1	<0.6	Li	300	30

*See description of leachate preparation in summary. Electrode formed directly (no Parr bomb).

TABLE 35. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF BED REJECT MATERIAL RUN 4 (COLUMN) (μg/1)

U	<6	Tb	<1	Ru	<15	V	5
Th	<6	Gd	<4	Mo	70	Ti	300
Bi	<1	Eu	<4	Nb	<0.6	Sc	<3
Pb	15	Sm	<6	Zr	3	Ca	500,000
Tl	<4	Nd	<2	Y	<0.6	K	10,000
Hg	<5	Pr	<1	Sr	700	Cl	2,000
Au	<5	Ce	<1	Rb	150	S	1,500,000
Pt	<5	La	70	Br	70	P	150
Ir	<2	Ba	500	Se	15	Si	400
Os	<5	Cs	7	As	150	Al	150
Re	<2	I	0.6	Ge	<4	Mg	200
W	5	Te	<4	Ga	3	Na	2,000
Ta	<100	Sb	<0.7	Zn	<100	F	<4
Hf	<4	Sn	100	Cu	30	B	200
Lu	<1	In	<2	Ni	300	Be	<0.06
Yb	<4	Cd	<2	Co	<10	Li	200
Tm	<1	Ag	<1	Fe	2,000		
Er	<4	Pd	<2	Mn	10		
Ho	<1	Rh	<2	Cr	30		
Dy	<4						

*Electrode formed directly (no Parr bomb).

TABLE 36. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF BED REJECT MATERIAL RUN 5 (ULTRASONIC SHAKING) (μg/l)

	Shake Number			Shake Number	
	1	10		1	10
U	<3	<3	Pd	<1	<1
Th	<3	<3	Rh	<1	<1
Bi	<0.5	<0.5	Ru	<5	<3
Pb	3	3	Mo	50	10
Tl	<1	<1	Nb	<0.3	<0.3
Hg	<2	<2	Zr	5	5
Au	<2	<2	Y	7	<0.3
Pt	<2	<2	Sr	700	700
Ir	<1	<1	Rb	50	50
Os	<2	<2	Br	4	5
Re	<1	<1	Se	15	25
W	<2	<2	As	40	50
Ta	≤70	≤150	Ge	<2	<2
Hf	<2	<2	Ga	5	1
Lu	<0.5	<0.5	Zn	<50	<50
Yb	<2	<2	Cu	20	15
Tm	<0.5	<0.5	Ni	50	70
Er	<2	<2	Co	<5	<5
Ho	<0.5	<0.5	Fe	700	1,000
Dy	<2	<2	Mn	5	10
Tb	<0.5	<0.5	Cr	50	100
Gd	<2	<2	V	100	15
Eu	<3	<2	Ti	150	500
Sm	<3	<3	Sc	<1.5	<2
Nd	<1.5	<2	Ca	400,000	150,000
Pr	<0.5	<0.4	K	2,500	7,000
Ce	0.7	0.5	Cl	300	700
La	0.7	<0.4	S	200,000	300,000
Ba	500	250	P	150	150
Cs	2.5	2	Si	25,000	2,500
I	0.5	0.5	Al	1,500	2,500
Te	<1.5	<2	Mg	1,000	5,000
Sb	<0.5	1	Na	1,500	1,500
Sn	5	1.5	F	<1.5	<2
In	<0.5	<3	B	15	15
Cd	<1.5	<2	Be	0.1	0.07
Ag	<1	1.5	Li	100	100

*See description of leachate preparation in summary. Electrode formed directly (no Parr bomb).

TABLE 37. SPARK SOURCE MASS SPECTROSCOPY
LEACHATE* OF BED REJECT MATERIAL
RUN 5 (COLUMN)
(μg/1)

U	<5	Tb	<1	Ru	<15	V	30
Th	<5	Gd	<3	Mo	400	Ti	1,000
Bi	<1	Eu	<5	Nb	<0.5	Sc	<3
Pb	20	Sm	<5	Zr	30	Ca	1,000,000
Tl	<2	Nd	<2	Y	<0.5	K	10,000
Hg	<4	Pr	<0.7	Sr	300	Cl	2,000
Au	<4	Ce	1	Rb	300	S	1,500,000
Pt	<4	La	40	Br	300	P	500
Ir	<2	Ba	700	Se	100	Si	1,500
Os	<4	Cs	20	As	150	Al	1,500
Re	<2	I	4	Ge	<4	Mg	70
W	20	Te	<3	Ga	3	Na	3,000
Ta	≤100	Sb	<0.6	Zn	<100	F	5
Hf	<3	Sn	10	Cu	100	B	300
Lu	<1	In	≤2	Ni	500	Be	1.5
Yb	<3	Cd	≤2	Co	0	Li	1,000
Tm	≤1	Ag	10	Fe	2,000		
Er	≤3	Pd	<6	Mn	10		
Ho	<1	Rh	<6	Cr	200		
Dy	<3						

*Electrode formed directly (no Parr bomb).

TABLE 38. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF PFBC CYCLONE NUMBER 2 DUST RUN 2 (ULTRASONIC SHAKING)

(μg/l)

	Shake Number			Shake Number	
	1	10		1	10
U	<3	<1	Pd	<1	<1
Th	<3	<1	Rh	<4	<6
Bi	<0.6	<0.2	Ru	<6	<7
Pb	4	1.5	Mo	100	60
Tl	<2	<0.4	Nb	0.4	≤0.6
Hg	<3	<1	Zr	6	2
Au	≤3	<1	Y	2	1
Pt	≤3	<1	Sr	4,000	1,500
Ir	<2	<0.4	Rb	400	200
Os	<3	<1	Br	4	10
Re	<2	<0.4	Se	60	200
W	10	<1	As	25	6
Ta	≤100	≤6	Ge	<3	<1
Hf	<2	<0.6	Ga	3	0.6
Lu	<0.6	<0.2	Zn	<60	<20
Yb	<2	<0.6	Cu	60	20
Tm	<0.6	<0.2	Ni	30	60
Er	<2	<0.	Co	<6	<6
Ho	<0.6	<0.2	Fe	600	40
Dy	<2	<0.	Mn	6	6
Tb	<0.6	<0.2	Cr	20	10
Gd	<2	<0.	V	100	40
Eu	<3	<0.	Ti	200	<20
Sm	<4	<1	Sc	<2	<0.6
Nd	<2	<0.	Ca	400,000	60,000
Pr	<0.6	<0.2	K	10,000	20,000
Ce	2	0.	Cl	100	100
La	2	0.2	S	400,000	100,000
Ba	600	60	P	200	10
Cs	4	0.	Si	4,000	400
I	2.5	0.	Al	2,000	30
Te	2	<0.	Mg	100,000	50,000
Sb	2.5	2	Na	3,000	6,000
Sn	6	<1	F	<2	<0.6
In	<0.6	<0.2	B	60	600
Cd	<2	≤1	Be	0.1	<0.03
Ag	≤1	<0.	Li	2,000	200

*See description of lachate preparation in summary. Electrode formed directly (no Parr bomb).

TABLE 39. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF PFBC CYCLONE NUMBER 2 DUST RUN 2 (COLUMN) (μg/1)

U	<15	Tb	<3	Ru	<15	V	250
Th	<15	Gd	<10	Mo	20	Ti	500
Bi	<3	Eu	<4	Nb	<2	Sc	<10
Pb	20	Sm	<15	Zr	25	Ca	700,000
Tl	<6	Nd	<6	Y	<2	K	25,000
Hg	<10	Pr	<2	Sr	4,000	Cl	2,000
Au	<10	Ce	2	Rb	150	S	2,000,000
Pt	<10	La	<2	Br	15	P	400
Ir	<6	Ba	2,000	Se	40	Si	15,000
Os	<10	Cs	5	As	70	Al	15,000
Re	<6	I	15	Ge	<10	Mg	5,000
W	7	Te	<6	Ga	15	Na	5,000
Ta	≤70	Sb	<2	Zn	<250	F	<10
Hf	≤10	Sn	7	Cu	70	B	15
Lu	<3	In	<6	Ni	150	Be	<0.15
Yb	<10	Cd	<6	Co	<30	Li	500
Tm	<3	Ag	<3	Fe	2,500		
Er	<10	Pd	<6	Mn	15		
Ho	<3	Rh	<6	Cr	130		
Dy	<10						

*Electrode formed directly (no Parr bomb).

TABLE 40. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF PFBC CYCLONE NUMBER 2 DUST RUN 4 (ULTRASONIC SHAKING)
(μg/1)

	Shake Number			Shake Number	
	1	10		1	10
U	<2	<2	Pd	<0.6	<0.7
Th	<2	<2	Rh	<1	<0.7
Bi	<0.3	<0.3	Ru	<2	<2
Pb	2	5	Mo	20	7
Tl	<0.6	<0.7	Nb	1	0.2
Hg	<2	<2	Zr	4	2
Au	<2	<2	Y	1.5	0.5
Pt	<2	<2	Sr	1,500	1,000
Ir	<0.6	<0.7	Rb	150	70
Os	<2	<2	Br	2	2
Re	<0.6	<0.7	Se	15	5
W	<1	≤1	As	20	10
Ta	≤50	≤30	Ge	<1.5	<2
Hf	<1	<1	Ga	6	2
Lu	<0.3	<0.3	Zn	<30	<30
Yb	<1	<1	Cu	10	15
Tm	<0.3	<0.3	Ni	30	50
Er	<1	<1	Co	<3	<4
Ho	<0.3	<0.3	Fe	1,500	700
Dy	<1	<1	Mn	7	3
Tb	<0.3	<0.3	Cr	30	30
Gd	<1	<1	V	60	30
Eu	<1	<2	Ti	150	100
Sm	<2	<2	Sc	3	1
Nd	2	<1	Ca	200,000	100,000
Pr	0.3	<0.3	K	6,000	5,000
Ce	1.5	0.7	Cl	60	250
La	1	0.3	S	200,000	250,000
Ba	300	300	P	70	50
Cs	3	3	Si	7,000	5,000
I	2	0.7	Al	7,000	1,500
Te	<1	<0.7	Mg	1,500	1,500
Sb	≤0.6	0.7	Na	3,000	1,500
Sn	3	1	F	<1	1.5
In	<0.3	<0.7	B	60	7
Cd	<1	<0.7	Be	0.15	0.02
Ag	1	0.3	Li	600	70

*See description of leachate preparation in summary. Electrode formed directly (no Parr bomb).

TABLE 41. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF PFBC CYCLONE NUMBER 2 DUST RUN 4 (COLUMN) (μg/l)

U	<3	Tb	<0.5	Ru	<3	V	20
Th	<3	Gd	<2	Mo	30	Ti	150
Bi	<0.5	Eu	<2	Nb	<0.3	Sc	<2
Pb	7	Sm	<3	Zr	3	Ca	500,000
Tl	<1	Nd	1	Y	<0.3	K	150,000
Hg	<2	Pr	<0.5	Sr	300	Cl	300
Au	<2	Ce	2	Rb	300	S	700,000
Pt	<2	La	10	Br	7	P	250
Ir	<1	Ba	100	Se	20	Si	5,000
Os	<2	Cs	20	As	15	Al	700
Re	<1	I	2	Ge	<2	Mg	1,500
W	1.5	Te	<2	Ga	2	Na	15,000
Ta	≤15	Sb	<0.3	Zn	<50	F	2.5
Hf	<2	Sn	3	Cu	50	B	100
Lu	<0.5	In	<1	Ni	30	Be	<0.01
Yb	<0.5	Cd	<1	Co	<5	Li	1,000
Tm	<0.5	Ag	0.5	Fe	200		
Er	<2	Pd	<1	Mn	2.5		
Ho	<0.5	Rh	<1	Cr	50		
Dy	<2						

*Electrode formed directly (no Parr bomb).

TABLE 42. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF PFBC CYCLONE NUMBER 2 DUST RUN 5 (ULTRASONIC SHAKING) (μg/1)

	Shake Number			Shake Number	
	1	10		1	10
U	<2	<2	Pd	<0.6	<0.6
Th	<2	<2	Rh	<2	<0.6
Bi	<0.3	<0.3	Ru	<3	<2
Pb	2	7	Mo	50	7
Tl	<0.5	<0.6	Nb	<0.4	0.2
Hg	<1	<1	Zr	3	4
Au	<1	<1	Y	0.4	0.7
Pt	<1	<1	Sr	1,000	1,500
Ir	<0.5	<0.5	Rb	50	150
Os	<1	<1	Br	4	1.5
Re	<0.5	<0.5	Se	30	<10
W	1.5	1	As	15	25
Ta	≤40	<15	Ge	<1	<1
Hf	<1	<1	Ga	10	10
Lu	<0.3	<0.3	Zn	<30	<30
Yb	<1	<1	Cu	15	20
Tm	<0.3	<0.3	Ni	40	40
Er	<1	<1	Co	<3	<3
Ho	<0.3	<1	Fe	300	2,500
Dy	<1	<1	Mn	7	10
Tb	<0.3	<0.3	Cr	70	50
Gd	<1	<1	V	150	100
Eu	<2	<2	Ti	150	250
Sm	<2	<2	Sc	<1	3
Nd	<1	4	Ca	250,000	250,000
Pr	<0.3	0.5	K	4,000	700
Ce	0.7	5	Cl	50	100
La	0.7	2	S	150,000	200,000
Ba	400	400	P	70	150
Cs	1	3	Si	7,000	15,000
I	1.5	0.6	Al	15,000	15,000
Te	<1	<0.5	Mg	1,000	4,000
Sb	<0.3	<0.3	Na	700	3,000
Sn	3	1	F	1.5	1.5
In	<0.3	<0.5	B	100	5 -
Cd	<1	<1	Be	0.04	0.04
Ag	<0.6	0.4	Li	250	50

*See description of leachate preparation in summary. Electrode formed directly (no Parr bomb).

TABLE 43. SPARK SOURCE MASS SPECTROSCOPY LEACHATE* OF PFBC CYCLONE NUMBER 2 DUST RUN 5 (COLUMN) (μg/1)

U	<3	Tb	<.5	Ru	<4	V	150
Th	<3	Gd	<2	Mo	100	Ti	150
Bi	<0.5	Eu	<2	Nb	<0.3	Sc	<2
Pb	7	Sm	<2	Zr	5	Ca	300,000
Tl	<1	Nd	<2	Y	<0.3	K	30,000
Hg	<2	Pr	<0.4	Sr	3,000	Cl	200
Au	<2	Ce	0.5	Rb	500	S	300,000
Pt	<2	La	2.5	Br	15	P	700
Ir	<1	Ba	150	Se	50	Si	2,500
Os	<2	Cs	10	As	20	Al	2,500
Re	<1	I	1	Ge	<2	Mg	10,000
W	2.5	Te	<1	Ga	20	Na	10,000
Ta	≤50	Sb	2	Zn	<50	F	15
Hf	<2	Sn	≤1.5	Cu	30	B	50
Lu	<0.5	In	≤1	Ni	30	Be	<0.03
Yb	<2	Cd	<7	Co	<5	Li	2,500
Tm	<0.5	Ag	2	Fe	200		
Er	<2	Pd	1	Mn	5		
Ho	<0.5	Rh	<3	Cr	150		
Dy	<2						

*Electrode formed directly (no Parr bomb).

TABLE 44. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS SOLID SAMPLES (μg/g)

Sample	Run	Hg*	Sb†	As‡
Feed coal	2	.17	<0.5	14
	5	.12	<0.5	7.5
Dolomite sorbent	2	<0.02	<0.5	<1
	5	<0.02	<0.5	2.2
Bed reject material	2	<0.02	<0.5	52
	4	<0.02	2.0	35
	5	<0.02	0.5	21
PFBC cyclone #2 Dust	2	<0.02	1.3	36
	4	0.05	2.0	33
	5	0.04	2.7	34

*Hg by flameless AAS.
†Sb by AAS with graphite furnace.
‡As by colorimetric/silver diethyl dithiocarbamate.

TABLE 45. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS SASS TRAIN SAMPLE (μg/g)

Sample	Run	Hg*	Sb†	As‡
Filter and first cyclone (<1 - 3μ particles)	2	<0.02	5.7	128
	4	0.16	5.3	85
	5	<0.02	4.0	45
Second and third cyclones (3 - 10μ particles)	2	0.69	3.7	83
	4	<0.02	3.3	64
	5	<0.02	2.3	36
XAD-2 resin	1	1.2	§	0.04
	2	§	149	0.15
	3	0.4	249	0.21
	4	0.3	60	0.15
	5	1.5	§	<0.05
First, second, and third impingers	2	~1.26	≲1.5	~2.1
	4	~1.27	≲1.2	~1.8
	5	0.50	≲1.5	≲0.8

*Hg by flameless AAS.
†Sb by AAS with graphite furnace.
‡As by colorimetric/silver diethyl dithiocarbamate.
§Not detected above level of blank.

TABLE 46. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS
($\mu g/m^3$ of gas sampled)*

Sample	Run	Hg†	Sb‡	As§
XAD-2 module condensate (acidified)	1	0.22	<0.1	0.05
	2	0.50	<0.3	0.1
	3	0.43	<0.2	0.16
	4	0.24	<0.1	0.17
	5	0.35	<0.2	0.35
XAD-2 module condensate (basified)	1	0.22	<0.1	<0.05
	2	0.50	<0.3	<0.1
	3	0.52	<0.2	0.08
	4	0.27	<0.1	0.11
	5	0.35	<0.2	0.35

*Each value represents the total concentrations in the gas phase based on separate analyses of the acidified and basified condensate solutions.
†Hg by flameless AAS.
‡Sb by AAS with graphite furnace.
§As by colorimetric/silver diethyl dithiocarbamate.

TABLE 47. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS
LEACHATE FROM BED REJECT MATERIAL*
($\mu g/l$)

Sample	Run	Hg†	Sb‡	As§
#1	2	<5	<20	50
#10		<5	<20	<50
Column		<5	<20	50
#1	4	<5	<20	110
#10		<5	<20	<50
Column		<5	<20	50
#1	5	<5	<20	300
#10		<5	<20	<50
Column		<5	<20	<50

*Artificial Leaching by ultrasonic shaking identified as shake #1 and #10. Artificial Leaching by Column identified as Column.
†Hg by flameless AAS.
‡Sb by AAS with graphite furnace.
§As by colorimetric/silver diethyl dithiocarbamate.

TABLE 48. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS LEACHATE FROM PFBC CYCLONE NUMBER 2 DUST

(μg/1)

Sample	Run	Hg*	Sb†	As‡
#1	2	<5	<20	70
#10		<5	<20	50
Column		<5	<20	50
#1	4	<5	<20	250
#10		<5	<20	50
Column		<5	<20	50
#1	5	<5	<20	50
#10		<5	<20	50
Column		<5	<20	<50

*Hg by flameless AAS.
†Sb by AAS with graphite furnace.
‡As by colorimetric/silver diethyl dithiocarbamate.

TABLE 49. GAS CHROMATOGRAPHY* FOR INORGANIC GASES FLUE GAS BEFORE AIR DILUTION

Sample	Run	SO_2 (ppm)	CO (ppm)	O_2%	CO_2%
	1	355	100	5.6 (15.5)	13.7
	2	151	130	7.6 (16.7)	9.8
	3	62	90	6.1 (15.5)	12.0
	4	41	56	6.0 (15.4)	12.5
	5	29	53	5.5 (15.3)	13.1

*Gas chromatography was not used to identify these gases. Continuous monitors already on line were used as indicated below:

SO_2 by IR (0-3000 ppm) or UV (0-100 ppm);
CO by NDIR;
O_2 by Beckman 715 Pt electrode; data in parentheses are O_2 concentrations after dilution;
CO_2 by IR.

TABLE 50. GAS CHROMATOGRAPHY* FOR INORGANIC GASES FLUE GAS BEFORE AIR DILUTION (ppm)

Run	SO_2	H_2S/COS	NH_3	HCN	F	Cl
1	583.2	---	0.06	<0.0008	7.3	47.1
2	156.2	<0.1	0.09	0.001	6.7	46.2
3	46.8	<0.1	0.27	<0.0004	12.3	42.5
4	70.2	<0.1	0.07	<0.0004	14.5	38.6
5	28.1	<6	0.64	0.001	12.8	33.5

*Gas chromatography was used to determine H_2S/COS after collection by evacuated bulb. The following were collected by midget impingers and analyzed as indicated:

SO_2 by barium perchlorate/thorin titration,
NH_3 by microkjeldahl titration,
HCN by colorimetric/pyrindine-pyrazolone,
F by ion chromatography,
Cl by gravimetric/silver chloride.

TABLE 51. GAS CHROMATOGRAPHY* FOR INORGANIC GASES FLUE GAS AFTER DILUTION

Sample	Run	O_2%	CO_2%
After air dilution (sample port 1a)	1	15.3	4.7
	2	15.0	5.0
	3	14.0	5.9
	4	No data	No data
	5	13.8	6.1
Before air dilution (sample port 1p)	1	No data	No data
	2	No data	No data
	3	5.9	12.8
	4	5.7†	12.1
	5	4.8	13.4
Dilution air (sample port 14)	1	20.1	0
	2	19.8	0
	3	21.0	0
	4	19.1	0
	5	No data	No data

*O_2/CO_2 by Orsat.
†Analyzed by Exxon.

TABLE 52. CHEMILUMINESCENCE FOR NO_x* FLUE GAS BEFORE AIR DILUTION

Sample	Units	NOx concentrations
Run 1	ppm	102 (15.0)†
Run 2	ppm	136 (29.0)†
Run 3	ppm	128 (40.0)†
Run 4	ppm	124 (no data)†
Run 5	ppm	119 (70.0)†

*Sampled from port 1p (after PFBC cyclone #2).
†Phenoldisulfonic acid method (evacuated bulb sample).

TABLE 53. ANION ANALYSIS OF COAL
(wt percent)

Run	Cl	F	SO_4	SO_3	NO_3	NO_2	CO_3	S
2	0.39	0.011	.04	*	<0.001	0.001	.10	1.10
5	0.59	0.011	.02	*	<0.001	<0.001	.10	1.03

*Not determined.
Analytical techniques:
Reduced sulfurs by ASTM D-2494-68
Cl -- Gravimetric/silver chloride
F -- Ion chromatography
NO_3 -- Ion chromatography
NO_2 -- Colorimetric/azo dye
CO_3 -- CO_2 evolution/gravimetric

TABLE 54. ANION ANALYSIS OF DOLOMITE SORBENT
(wt percent)

Run	Cl	F	SO_4	SO_3	NO_3	NO_2	CO_3	S
2	0.054	<0.001	0.7	0.003	<0.001	0.002	63.8	0.006
5	0.050	0.011	0.5	0.003	<0.001	<0.001	63.0	0.13

Analytical techniques:
Cl -- Gravimetric/silver chloride
F -- Distillation/ion chromatography
SO_4 -- Gravimetric/barium sulfate
SO_3 -- SO_2 evolution/colorimetric (p-rosaniline)
NO_3 -- Ion chromatography
NO_2 -- Colorimetric/azo dye
CO_3 -- CO_2 evolution/gravimetric
S -- H_2S evolution/titration

TABLE 55. ANION ANALYSIS OF PARTICULATE EMISSIONS--SASS COLLECTION (wt percent)

	Run	Cl	F	SO_4	SO_3	NO_3	NO_2	CO_3	S
<3μ	2	0.10	0.015	12.1	0.002	0.004	<0.001	<0.2	0.03
	4	0.002	0.032	10.8	0.002	0.001	<0.001	<0.2	<0.03
	5	0.011	0.031	9.4	0.001	<0.001	<0.001	<0.2	<0.03
>3μ	2	0.097	0.014	10.4	0.003	0.002	<0.001	<0.2	0.03
	4	0.005	0.032	10.1	0.003	0.003	<0.001	<0.2	<0.03
	5	0.007	0.032	8.7	0.004	<0.001	<0.001	<0.2	0.03

Analytical techniques:
Cl -- Colorimetric/ferricyanide method
All others same analyses as used on dolomite sorbent.

TABLE 56. ANION ANALYSIS OF BED REJECT MATERIAL (wt percent)

Run	Cl	F	SO_4	SO_3	NO_3	NO_2	CO_3	S
2	.026	.004	32.3	.012	0.004	<0.001	12.0	.014
4	.025	.016	26.1	.006	0.002	<0.001	8.7	.005
5	.030	.003	27.7	.011	0.001	<0.001	15.1	.005

Analyses same as used on dolomite sorbent.

TABLE 57. ANION ANALYSIS OF PFBC CYCLONE NUMBER 2 DUST (wt percent)

Run	Cl	F	SO_4	SO_3	NO_3	NO_2	CO_3	S
2	.024	.024	11.1	.015	<0.001	0.007	3.1	.010
4	.030	.017	14.2	.014	<0.001	0.006	2.3	.003
5	.031	.022	14.2	.024	<0.001	0.002	1.6	.005

Analyses same as used on dolomite sorbent.

TABLE 58. ANION ANALYSIS OF LEACHATES FROM BED REJECT MATERIAL*
(μg/l)

Sample		Cl	F	SO_4	SO_3	NO_3	NO_2
Run 2	#1	11,500	2,400	3.0×10^6	800	300	1,100
	#10	5,000	1,900	1.6×10^6	1,000	8,300	200
	column	176,000	4,000	1.5×10^6	3,100	<300	700
Run 4	#1	5,900	1,700	1.5×10^6	300	<300	1,500
	#10	2,200	300	1.3×10^6	100	<300	400
	column	64,000	2,100	0.2×10^6	1,500	<300	800
Run 5	#1	5,500	1,100	1.0×10^6	300	<300	<200
	#10	4,800	1,100	1.5×10^6	500	7,100	300
	column	62,000	1,700	0.2×10^6	1,300	1,800	1,900

*Artificial leaching by ultrasonic shaking identified as shake #1 and #10. Artificial leaching by column method identified as column.

TABLE 59. ANION ANALYSIS OF LEACHATES FROM PFBC CYCLONE NUMBER 2 DUST*
(μg/l)

Sample		Cl	F	SO_4	SO_3	NO_3	NO_2
Run 2	#1	6,700	2,400	2.5×10^6	300	6,400	8,600
	#10	7,100	2,300	0.8×10^6	500	6,700	300
	column	65,000	2,000	0.9×10^6	2,300	2,500	71,000
Run 4	#1	8,700	900	1.0×10^6	<100	1,300	8,600
	#10	200	200	1.2×10^6	100	<300	300
	column	53,000	400	0.2×10^6	1,500	<300	49,000
Run 5	#1	12,500	1,800	1.1×10^6	<100	1,600	3,300
	#10	5,800	300	1.1×10^6	<100	300	300
	column	33,000	1,200	0.2×10^6	1,300	3,000	17,000

*Artificial leaching by ultrasonic shaking identified as shake #1 and #10. Artificial leaching by column method identified as column.

TABLE 60. GAS CHROMATOGRAPHY FOR C_7-C_{17}

Sample	Range	Volatile weight, µg/g	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Feed coal - Run 2	GC7 90 - 110	389	†	†	†
	GC8 110 - 140	345			
	GC9 140 - 160	193			
	GC10 160 - 180	69			
	GC11 180 - 200	103			
	GC12 200 - 220	72			
	GC13 and greater	418			
Feed coal - Run 5	GC7 90 - 110	302			
	GC8 110 - 140	354			
	GC9 140 - 160	190			
	GC10 160 - 180	61			
	GC11 180 - 200	85			
	GC12 200 - 220	81			
	GC13 and greater	209			
		µg/m_3			
Flue gas* (diluted) Run 1	GC7 90 - 110	120			
	GC8 110 - 140	1170			
	GC9 140 - 160	0			
	GC10 160 - 180	384			
	GC11 180 - 200	31			
	GC12 200 - 220	7			
	GC13 and greater	86			

*Collected by XAD-2 module.
†Not reported.

TABLE 61. GAS CHROMATOGRAPHY FOR C_7-C_{17}

Sample	Range	Volatile weight, μg/m³	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Flue gas* (diluted) Run 2	GC7 90 - 110	1085	†	†	†
	GC8 110 - 140	1215			
	GC9 140 - 160	429			
	GC10 160 - 180	1037			
	GC11 180 - 200	527			
	GC12 200 - 220	478			
	GC13 and greater	3337			
Flue gas* (diluted) Run 3	GC7 90 - 110	34			
	GC8 110 - 140	654			
	GC9 140 - 160	0			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	29			
Flue gas* (diluted) Run 4	GC7 90 - 110	1058			
	GC8 110 - 140	1911			
	GC9 140 - 160	403			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	25			
	GC13 and greater	145			

*Collected by XAD-2 module.
†Not reported.

TABLE 62. GAS CHROMATOGRAPHY FOR C_7-C_{17}

Sample	Range	Volatile weight, μg/m^3	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Flue gas* (diluted) Run 5	GC7 90 - 110	110	†	†	†
	GC8 110 - 140	1630			
	GC9 140 - 160	0			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	58			
Diluent air Run 1	GC7 90 - 110	616			
	GC8 110 - 140	9086			
	GC9 140 - 160	0			
	GC10 160 - 180	1970			
	GC11 180 - 200	260			
	GC12 200 - 220	37			
	GC13 and greater	443			
Diluent air Run 2	GC7 90 - 110	819			
	GC8 110 - 140	1397			
	GC9 140 - 160	311			
	GC10 160 - 180	267			
	GC11 180 - 200	329			
	GC12 200 - 220	294			
	GC13 and greater	5482			

*Collected by XAD-2 module.
†Not reported.

TABLE 63. GAS CHROMATOGRAPHY FOR C_7-C_{17} FLUE GAS PARTICULATES <3μ

Sample	Range	Volatile weight, μg/g	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Run 2	GC7 90 - 110	63	*	*	*
	GC8 110 - 140	113			
	GC9 140 - 160	116			
	GC10 160 - 180	35			
	GC11 180 - 200	46			
	GC12 200 - 220	25			
	GC13 and greater	18			
Run 4	GC7 90 - 110	659			
	GC8 110 - 140	1038			
	GC9 140 - 160	266			
	GC10 160 - 180	65			
	GC11 180 - 200	118			
	GC12 200 - 220	88			
	GC13 and greater	81			
Run 5	GC7 90 - 110	56			
	GC8 110 - 140	68			
	GC9 140 - 160	32			
	GC10 160 - 180	9			
	GC11 180 - 200	12			
	GC12 200 - 220	10			
	GC13 and greater	7			

*Not reported.

TABLE 64. GAS CHROMATOGRAPHY FOR C_7-C_{17} FLUE GAS PARTICULATES >3µ

Sample	Range	Volatile weight, µg/g	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Run 2	GC7 90 - 110	0	*	*	*
	GC8 110 - 140	340			
	GC9 140 - 160	193			
	GC10 160 - 180	415			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	265			
Run 4	GC7 90 - 110	69			
	GC8 110 - 140	4			
	GC9 140 - 160	12			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	1			
Run 5	GC7 90 - 110	22			
	GC8 110 - 140	30			
	GC9 140 - 160	17			
	GC10 160 - 180	5			
	GC11 180 - 200	8			
	GC12 200 - 220	6			
	GC13 and greater	6			

*Not reported.

TABLE 65. GAS CHROMATOGRAPHY FOR C_7-C_{17} SASS FRONT HALF WASH

Sample	Range	Volatile weight, μg/g	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Run 2	GC7 90 - 110	0	*	*	*
	GC8 110 - 140	23,368			
	GC9 140 - 160	0			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	236			
Run 4	GC7 90 - 110	0			
	GC8 110 - 140	732			
	GC9 140 - 160	0			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	11			
Run 5	GC7 90 - 110	0			
	GC8 110 - 140	195			
	GC9 140 - 160	0			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	5			

*Not reported.

TABLE 66. GAS CHROMATOGRAPHY FOR C_7-C_{17}
BED REJECT MATERIAL

Sample	Range	Volatile weight, µg/g	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Run 2	GC7 90 - 110	14	*	*	*
	GC8 110 - 140	16			
	GC9 140 - 160	6			
	GC10 160 - 180	13			
	GC11 180 - 200	7			
	GC12 200 - 220	6			
	GC13 and greater	43			
Run 4	GC7 90 - 110	0			
	GC8 110 - 140	0			
	GC9 140 - 160	0			
	GC10 160 - 180	0			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	0			
Run 5	GC7 90 - 110	20			
	GC8 110 - 140	22			
	GC9 140 - 160	12			
	GC10 160 - 180	4			
	GC11 180 - 200	5			
	GC12 200 - 220	3			
	GC13 and greater	17			

*Not reported.

TABLE 67. GAS CHROMATOGRAPHY FOR C_7-C_{17} PFBC CYCLONE NUMBER 2 DUST

Sample	Range	Volatile weight, μg/g	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Run 2	GC7 90 - 110	117	*	*	*
	GC8 110 - 140	9			
	GC9 140 - 160	0			
	GC10 160 - 180	6			
	GC11 180 - 200	0			
	GC12 200 - 220	0			
	GC13 and greater	0.3			
Run 4	GC7 90 - 110	15			
	GC8 110 - 140	19			
	GC9 140 - 160	10			
	GC10 160 - 180	3			
	GC11 180 - 200	4			
	GC12 200 - 220	3			
	GC13 and greater	3			
Run 5	GC7 90 - 110	41			
	GC8 110 - 140	53			
	GC9 140 - 160	31			
	GC10 160 - 180	8			
	GC11 180 - 200	10			
	GC12 200 - 220	7			
	GC13 and greater	17			

*Not reported.

TABLE 68. LC FRACTIONATION*
METHYLENE CHLORIDE EXTRACT OF FEED COAL

	Run 2				Run 5			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		161				82		
LC 2		85				105		
LC 3		198				177		
LC 4		82				104		
LC 5		2				83		
LC 6		306				335		
LC 7		664				421		
LC 8		0				0		

*All LC fractionations expressed as weight per gram of sample--corrected for solvent blanks.

TABLE 69. LC FRACTIONATION
FLUE GAS*

	Run 1				Run 2			
Fraction	TCO	Grav µg/Nm^3	Total	Total	TCO	Grav µg/Nm^3	Total	Total
LC 1		67				4104		
LC 2		71				202		
LC 3		32				944		
LC 4		18				283		
LC 5		41				324		
LC 6		367				1337		
LC 7		†				157		
LC 8		1802				749		

*Collection by SASS XAD-2 module and condenser (pentane extraction) and combined for organic analysis.
†Residue dissolved weighing dish.

TABLE 69 (con.)

	Run 3				Run 4			
Fraction	TCO	Grav µg/Nm^3	Total	Total	TCO	Grav µg/Nm^3	Total	Total
LC 1		60				314		
LC 2		2				32		
LC 3		2				11		
LC 4		66				830		
LC 5		148				369		
LC 6		67				714		
LC 7		*				290		
LC 8		371				978		

	Run 5			
Fraction	TCO	Grav µg/Nm^3	Total	Total
LC 1		58		
LC 2		107		
LC 3		238		
LC 4		86		
LC 5		106		
LC 6		636		
LC 7		159		
LC 8		407		

*Residue dissolved weighing dish.

TABLE 70. LC FRACTIONATION
DILUENT AIR*

	Run 1				Run 2			
Fraction	TCO	Grav µg/Nm^3	Total	Total	TCO	Grav µg/Nm^3	Total	Total
LC 1		2214				1835		
LC 2		29				11		
LC 3		17				27		
LC 4		33				53		
LC 5		55				119		
LC 6		297				133		
LC 7		586				537		
LC 8		9080				6184		

*Collection at sample port 14 by Tenax sorbent.

TABLE 71. LC FRACTIONATION
METHYLENE CHLORIDE EXTRACT OF FLUE GAS PARTICULATES
(> 3μ)

	Run 2				Run 4			
Fraction	TCO	Grav μg/g	Total	Total	TCO	Grav μg/g	Total	Total
LC 1		7				16		
LC 2		4				27		
LC 3		33				44		
LC 4		0				0		
LC 5		8				0		
LC 6		238				0		
LC 7		926				0		
LC 8		0				0		

	Run 5			
Fraction	TCO	Grav μg/g	Total	Total
LC 1		0		
LC 2		0		
LC 3		13		
LC 4		28		
LC 5		17		
LC 6		35		
LC 7		0		
LC 8		0		

TABLE 72. LC FRACTIONATION METHYLENE CHLORIDE EXTRACT OF FLUE GAS PARTICULATES (<3 μ)

	Run 2				Run 4			
Fraction	TCO	Grav μg/g	Total	Total	TCO	Grav μg/g	Total	Total
LC 1		0				767		
LC 2		33				494		
LC 3		39				461		
LC 4		27				28		
LC 5		122				0		
LC 6		103				0		
LC 7		139				0		
LC 8		0				562		

	Run 5			
Fraction	TCO	Grav μg/g	Total	Total
LC 1		0		
LC 2		14		
LC 3		43		
LC 4		91		
LC 5		0		
LC 6		44		
LC 7		0		
LC 8		0		

TABLE 73. LC FRACTIONATION
SASS FRONT HALF WASH

	Run 2				Run 4			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		78				42		
LC 2		15				4		
LC 3		2				38		
LC 4		10				19		
LC 5		348				4		
LC 6		710				143		
LC 7		426				0		
LC 8		22,015				493		

	Run 5			
Fraction	TCO	Grav µg/g	Total	Total
LC 1		22		
LC 2		2		
LC 3		14		
LC 4		27		
LC 5		135		
LC 6		0		
LC 7		0		
LC 8		0		

TABLE 74. LC FRACTIONATION METHYLENE CHLORIDE EXTRACT OF PFBC CYCLONE NUMBER 2 DUST

	Run 2				Run 4			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		0				0		
LC 2		73				12		
LC 3		29				0		
LC 4		1				22		
LC 5		18				23		
LC 6		11				0		
LC 7		0				0		
LC 8		0				0		

	Run 5			
Fraction	TCO	Grav µg/g	Total	Total
LC 1		0		
LC 2		0		
LC 3		0		
LC 4		0		
LC 5		0		
LC 6		63		
LC 7		104		
LC 8		0		

TABLE 75. LC FRACTIONATION
METHYLENE CHLORIDE EXTRACT OF BED REJECT MATERIAL

	Run 2				Run 4			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		0				0		
LC 2		0				0		
LC 3		0				0		
LC 4		0				0		
LC 5		0				0		
LC 6		34				0		
LC 7		70				0		
LC 8		0				0		

	Run 5			
Fraction	TCO	Grav µg/g	Total	Total
LC 1		0		
LC 2		0		
LC 3		0		
LC 4		<1		
LC 5		6		
LC 6		64		
LC 7		0		
LC 8		0		

IR ANALYSIS OF LC FRACTIONS

NOTE:

1. Species reported are those which are clearly different from species observed in the solvent, XAD-2, and Tenax blanks.

2. Fractions seven and eight of many samples contained water that presented great difficulty in the IR analysis. It was impossible to eliminate the water with moderate heating; thus, it is believed to be water of hydration of inorganic compounds in the fractions.

3. Clean denotes at or below IR detection limits of 1 to 5 μg.

TABLE 76. IR REPORT
SAMPLE: BEFORE LC SEPARATION

Sample	Run	Findings
Feed Coal	2	Diethylene glycol and probably water and carboxylic acid.
	5	Diethylene glycol, water, and carboxylic acid.
XAD-2	1	Water and aromatic sulfonate.
	2	Silicones, ester, and carboxylic acid.
	3	Aliphatic HC, water, and ethers.
	4	Aliphatic HC and ester carbonyl.
	5	High aliphatic HC content with mixture of carbonyl species, including ester, phthalates, and ethers.
Dilution Air	1	Virtually pure aliphatic HC, probable traces of carbonate and sulfate.
	2	Same as Run 1.
SASS 10μ + 3μ particulates	2	Large amount of diethylene glycol, some water and a carboxylic acid and its salt are also probably present.
	4	Large amount of diethylene glycol. Some esters and probably some acids are present which do not appear in the LC fractions.
	5	Strong ester and diethylene glycol bonds; however, not much ester appears in the fractions.
SASS 1μ + Filter Particulates	2	Silicones, ether, and carbonyls of ester and/or acids.
	4	Rather weak total spectrum, showing some carbonyl bonds which were observed in LC fractions 6 and 7. More of the 1740 cm^{-1} ester carbonyl is seen in the unseparated sample than in the LC fractions.
	5	Small quantity of material, only inorganics and some carbonyl (ester of ketone).
SASS Front Half Wash	2	Small amount of silicones, aromatic sulfonate, ester, and probably carboxylic acid.
	4	Primarily a hydrated sulfate. Water, but not sulfate, observed in LC fraction 8.
	5	Silicones, ester, and some carbonyl.

TABLE 76 (con.)

Sample	Run	Findings
FBC Bed Reject	2	Small amount of aliphatic HC and ester carbonyl.
	4	Weak spectrum, some diethylene glycol and ester carbonyl.
	5	Very weak spectrum showing a small quantity of diethylene glycol and some ester carbonyl.
FBC Cyclone Number 2 Dust	2	Small amount of aliphatic HC and ester carbonyl.
	4	Very weak spectrum, trace of carbonyl.
	5	Very clean, no functional groups observed.
Method 5 Filter	3	Primarily inorganics--carbonates and sulfates. Small amount of aliphatic HC.
Balston Filter	3	Small quantity of aliphatic HC and bands, probably resulting from water and a carboxylic acid and its salts.

TABLE 77. IR REPORT
SAMPLE: FEED COAL

LC	Run 2	Run 5
LC 1	Aliphatic HC	Aliphatic HC
LC 2	Aliphatic HC, aromatic HC	Aliphatic HC, aromatic HC
LC 3	Aromatic (fused ring?) HC	Aromatic (fused ring?) HC
LC 4	Aromatic HC (trace)	Aliphatic HC, ester, ketone, acid
LC 5	Clean	-----
LC 6	Diethylene glycol, mixed aromatic carbonyl	Phenol, carbonyl species
LC 7	Diethylene glycol	Diethylene glycol
LC 8	Diethylene glycol, water	Aromatic carboxylic acid, amide, water

TABLE 78. IR REPORT
SAMPLE: FLUE GAS (FROM XAD-2)

LC	Run 1	Run 2	Run 3	Run 4	Run 5
LC 1	Aliphatic HC	Aliphatic HC	Aliphatic HC	Aliphatic HC	Aliphatic HC
LC 2	Silicone	Silicone	(Clean)	(Clean)	Silicone
LC 3	Silicone	Silicone, tetradecaethyl siloxane	(Clean)	-----	Silicone, ester
LC 4	Silicone, ester	Silicone	Aromatic carboxylic acid, benzoic acid	Background	Ester
LC 5	Silicone, ester, aromatic structure	Silicone	Benzoic acid + ester, aliphatic carboxylic acid	-----	Background
LC 6	Carboxylic acid, dichlorobenzoic acid	Silicone, ester ketone	Aliphatic acid, ether	Background	Ether acetamide
LC 7	Sulfonic acid	Ester, ether, acid	Ether	Ether	Ester
LC 8	Aromatic sulfonate, water of hydration	Carboxylic acid + salt, chloride of sulfur	Aromatic sulfonate	-----	Polar material

TABLE 79. IR REPORT
SAMPLE: DILUENT AIR

LC	Run 1	Run 2
LC 1	Aliphatic HC	Aliphatic HC
LC 2	Clean	Clean
LC 3	Clean	Clean
LC 4	-----	-----
LC 5	-----	Ketone, ester
LC 6	Aromatic ketone, ester, phenol	Ester, ketone, phenol
LC 7	Water, ketone, aliphatic alcohol, NH_4Cl	Water, ketone, aliphatic alcohol, NH_4Cl
LC 8	Carboxylic acid, salts, water	Carboxylic acid, aromatic amine, phenol

TABLE 80. IR REPORT
SAMPLE: FLUE GAS PARTICULATES >3μ

LC	Run 2	Run 4	Run 5
LC 1	Silicone, aliphatic HC	----	Aliphatic HC, water
LC 2	Clean	Clean	----
LC 3	Ester	Clean	Clean
LC 4	Clean	Clean	Clean
LC 5	Clean	Clean	----
LC 6	Silicone	----	----
LC 7	Diethylene glycol	Ester	Diethylene glycol, ester
LC 8	Carboxylic acid, hydroxyl group	Carboxylic acid, ether, or alcohol	Ketone, ether

FLUE GAS PARTICULATES <3μ

LC	Run 2	Run 4	Run 5
LC 1	Aliphatic HC	Aliphatic HC, water	Aliphatic HC
LC 2	Clean	Clean	Clean
LC 3	Clean	Clean	Clean
LC 4	Ester, water, HC	Clean	Clean
LC 5	Aromatic HC, ester	Clean	Clean
LC 6	Carbonyls, amine, or hydroxyl	Carbonyls, ketone NH_4Cl	Carbonyls (ester or ketone)
LC 7	Ester, HC	Mix of carbonyls, carboxylic acid	Clean
LC 8	Carboxylic acid	Mix of carbonyls, carboxylic acid	Carboxylic acid, other polar materials

TABLE 81. IR REPORT
SAMPLE: SASS FRONT HALF WASH

LC	Run 2	Run 4	Run 5
LC 1	Background	Background	Aliphatic HC
LC 2	Clean	Aliphatic HC	Clean
LC 3	Silicone, ester	Trace HC + ester	Trace ester
LC 4	Silicone, ester	Trace ester	Phthalate + ester + aromatic + aldehyde
LC 5	Silicone, esters, aromatic structure	Ester + phthalate	Ketone + phthalate and/or ester + aromatic structure
LC 6	Ester alcohol + possible ethylene glycol monoacetate + other esters	Background	Ketone + Carboxylic acid + ester and/or phthalate
LC 7	Sulfate + water of hydration	Ester + possible carboxylic acid + salt	Trace carboxylic acid + acid salt + ester
LC 8	Carboxylic acid + acid salt	Carboxylic acid + acid salt + water of hydration	Water of hydration + phthalate + aromatic sulfonate

TABLE 82. IR REPORT
SAMPLE: BED REJECT MATERIAL

LC	Run 2	Run 4	Run 5
LC 1	Clean	Aliphatic HC, phthalate	Aliphatic HC, carbonyl species, (phthalate?)
LC 2	Clean	Phthalate	Carbonyl (phthalate?)
LC 3	Clean	Clean	Carbonyl (phthalate?)
LC 4	Clean	Clean	Alcohol, ester
LC 5	Clean	Clean	Ester, ketone
LC 6	Ketone	Ketone	Mix of carbonyls
LC 7	Clean	Diethylene glycol, ester, water	Diethylene glycol, mix of carbonyls
LC 8	Inorganics, NH_4Cl	Water	Water

TABLE 83. IR REPORT
SAMPLE: PFBC CYCLONE NUMBER 2 DUST

LC	Run 2	Run 4	Run 5
LC 1	Aliphatic HC	Aliphatic HC	Ester (acetate?)
LC 2	Clean	Aliphatic HC	Amide, urea types
LC 3	Clean	Aliphatic HC	Amide, urea types
LC 4	Clean	Aliphatic HC, carbonyl species	Amide, urea types
LC 5	Clean	Clean	Amide, urea types
LC 6	Amine, phenol	Ketone	Ketone
LC 7	Clean	Aliphatic HC, ester	Diethylene glycol
LC 8	Carboxylic acid	Unsaturated carboxylic acid, other polar species	Phthalate

TABLE 84. IR REPORT
SAMPLE: METHOD 5 FILTER

LC	Findings
LC 1	Aliphatic HC
LC 2	Clean
LC 3	Clean
LC 4	Clean
LC 5	Clean
LC 6	Ester + ketone + carboxylic acid
LC 7	Clean
LC 8	Aromatic sulfonate

TABLE 85. IR REPORT
SAMPLE: BALSTON FILTER

LC	Findings
LC 1	Clean
LC 2	Clean
LC 3	Clean
LC 4	Clean
LC 5	Clean
LC 6	Ester + ketone + carboxylic acid
LC 7	Ester + ketone + carboxylic acid
LC 8	Carboxylic acid + acid salt

TABLE 86. LRMS REPORT
SAMPLE: XAD-2 MODULE

1. **Categories Present**

Run	LC	Intensity	Category	MW Range
1	8	---	---	---
2	1	---	Aliphatic HC	---
	3	---	None	---
	6	---	None	---
	8	---	None	---
4	4	---	None	
	6	---	---	---
5	6	---	---	---

2. **Subcategories, Specific Compounds**

Run	LC	Intensity	Compound	m/e	Composition
2	1	100		---	$<C_{13}$
	3	100	Tetra-decaethyl siloxane	---	---
	6	100	Dichlorobenzoic acid	---	---
	8	None	None	---	---
4	4	None	None	---	---
	6	None	None	---	---
5	6	100	Acetamide?	---	---

3. **Other**

Intensity

N.R.

TABLE 87. LRMS REPORT
SAMPLE: SASS FRONT HALF WASH

1. Categories Present

Run	LC	Intensity	Category	MW Range
2	8	10	Chloride or sulfur	---
4	8	None	None	---

2. Subcategories, Specific Compounds

Run	LC	Intensity	Compound	m/e	Composition
2	8	None	None	---	---
4	8	None	None	---	---

3. Other

Intensity
N.R.

TABLE 88. LRMS REPORT
SAMPLE: PFBC DILUTION AIR

1. Categories Present

Run	LC	Intensity	Category	MW Range
1	8		None	None
2	8		None	None

2. Subcategories, Specific Compounds

Run	LC	Intensity	Compound	m/e	Composition
1	8	None	None	---	---
2	8	None	None	---	---

3. Other

Intensity
N.R.

STUDY NUMBER 11

DATA SOURCE:

COMPREHENSIVE ANALYSIS OF EMISSIONS FROM MERC FLUIDIZED-BED COMBUSTION UNIT

DATA STATUS: Draft Report, September, 1977

AUTHORS: J. M. Allen, E. L. Merryman, J. E. Howes, Jr., S. E. Miller, and E. J. Schulz

CONTRACTOR:
Battelle Columbus Laboratories
Columbus, Ohio 43201
Contract No. 68-02-2138

PROJECT OFFICER:
D. B. Henschel
Industrial Environmental Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

In September 1976, Battelle Columbus Laboratories (BCL) sampled the 18-inch atmospheric fluidized-bed combustor (FBC) at the Morgantown Energy Research Center (MERC). Atypical aspects of this operation were: use of a cone-shaped FBC distributor plate in the MERC unit; use of a lower ash, lower Btu anthracite culm as an FBC fuel (anthracite culm ≅5,000 Btu/lb; about one-half the value of normally used coal); no use of limestone sorbent in most test runs; lower velocity within the bed (~3 ft/sec); and use of poorly functioning bag filters in the gaseous effluent stream just prior to discharge. Figure 1 from the study diagrams the MERC operation. Fuel feed rate was 90 lb/hr, and bed temperature was ~1,600° F. The stated objective of this study was "to measure all potential pollutants from both direct and indirect sources, which might be of significance in the commercialization of this specific type atmospheric" FBC process.

Four sampling runs of 5 hours each were made. Runs 1, 3, and 4 were made under "normal" operating conditions. In run 2, fines from the first FBC cyclone were recycled into the bed and some limestone sorbent was added to the bed. Only samples from runs 2, 3, and 4 were analyzed. Nine streams, from a 40-stream list, were sampled. These were: stack gas from FBC, particulate removal discard from FBC, bed solids discard from FBC, fuel feed to FBC, flue gas from FBC to particle removal, recycle of particulates from particle removal to FBC (run 2 only), fugitive or secondary emission from FBC particulate disposal, and effluent gas from secondary stack gas cleaning device.

Some conclusions drawn in the report of this study were: that excess O_2 caused low CO (≤400 ppm) and low levels of reduced sulfur compounds; that sulfur compounds were more concentrated in the smaller particulate fractions; that the NO (average concentrations of 222 ppm) was probably from the fuel N; and that NO_2 from this process was very low (<5 ppm). A comparative evaluation of the MERC FBC with FBC units at Argonne National Laboratory (ANL) and Battelle Columbus Laboratories (BCL) showed an overall wide variation, perhaps due to use of different coals. However, there were strong tendencies for the 33 elements of interest to concentrate, going from larger to smaller particle size, and from bed ash to the first cyclone to the second cyclone. Considering the MERC and BCL FBC units in relation to the N and S levels in the different types of coal, the two units were roughly equivalent in emission of SO_2, SO_3, CN^- and Cl^-; the BCL unit was somewhat higher for NH_3; and the MERC unit was much higher for F^-. The BCL and MERC units were comparable for organic emissions in most cases within a factor of 5, excepting the BCL cyclones which were much higher in organics. The BCL FBC gaseous emissions were somewhat higher in As than the MERC. Bioassays showed no significant mutagenic activity, no significant bacterial toxicity, and no significant cytotoxicity.

The modified method 5 sampler (see subsequent description) showed par-

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ticulate loadings of: Run 2--7,320 mg/m^3, Run 3--2,940 mg/m^3, and Run 4--2,490 mg/m^3 at the bag collector inlet. The HVSS (subsequently described) showed loadings of Run 2--1,518 mg/m^3, Run 3--1,497 mg/m^3, and Run 4--1,014 mg/m^3 at the stack effluent, after the bag collectors.

GASEOUS GRAB

The specified glass bulb was not used for some gaseous grab sampling. Some samples were taken by continuous withdrawal to autoanalyzers (CO_2 and CO by NDIR; SO_2 by flame photometric; NO and NO_x by chemiluminescence; and O_2 by paramagnetic). Other gaseous samples were drawn with separate sampling trains. SO_3 and H_2SO_4 were sampled with a Göksyr-Ross controlled condensation apparatus and analyzed by ion chromatography. NH_3 was sampled with a modified Method 6 train containing H_2SO_4 in impingers and analyzed by the Kjeldahl method. Cyanides were sampled with a modified Method 6 train with KOH in impingers and analyzed by a colorimetric method. HCl was sampled with a modified Method 6 train with NaOH in impingers and analyzed by titration, and volatile fluorides were sampled with a Method 6- type train with NaOH in impingers and analyzed by specific ion electrode. CO_2, O_2, and CO were determined by Orsat from the evacuated bulb sample. The evacuated flask or bag technique was used to obtain samples for total hydrocarbon analysis at Battelle. H_2S, COS, and disulfides were sampled by an evacuated glass bulb and analyzed by gas chromatography-gas mass spectroscopy at Battelle.

SASS

A SASS train was not used in this study. A modified Method 5 train was used to collect particulates, organic vapors, and trace metals from the gaseous stream entering the bag collector. This modified Method 5 train consisted of a probe, two glass cyclones held at 250° F (cutoff sizes at 4.6µ and 2.1µ), a quartz filter at 250° F, a Tenax-GC trap held at 125° F, an impinger containing an H_2O_2 solution, an empty impinger, two more impingers containing 1 N HNO_3 (originally used $(NH_4)_2S_2O_8/AgNO_3$ solution, but encountered precipitate), an empty impinger, and a sixth impinger containing silica gel. All impingers were in an ice bath. A flow rate of 0.75 cfm was used during sampling. Sample was rinsed from the apparatus with CH_2Cl_2.

An HVSS was used to measure the effluent stream leaving the stack. This system was similar to the modified Method 5 train but with a heated probe at 250° F, heated filter at 250° F, and impingers in an ice bath. The first and second impingers contained water, the third was empty, and the fourth contained silica gel. The HVSS contained no cyclones or Tenax-GC trap. The HVSS system was operated at about 1.5 scfm. The HVSS was rinsed with acetone.

A Mark III, eight-stage, Andersen cascade impactor, connected to the modified Method 5 train probe and heated to 250° F by the method 5 train oven, was used to size particulates. A high-purity quartz substrate was the collecting medium.

The samples collected by the modified Method 5 train, HVSS, and cascade impactor were analyzed by SSMS for trace metals. Major elements were determined by emission spectroscopy. Organic analysis followed the usual Level I procedure of extraction, LC separation and IR, but no LRMS analysis was performed. Biological samples were assayed by the Ames' bacterial mutagenicity test, the mammalian cell cytotoxicity assay (prescreen confluency assay), and the bacterial toxicity assay (percent survival of the Ames' bacterial strains).

FUGITIVE EMISSIONS

Artificial leachates were prepared from the bed discard and the first and second FBC cyclone discards by two different procedures. In the first method, sample plus water was agitated ultrasonically for 10 1-hour increments; then the first and tenth leachate solutions were analyzed for trace metals (SSMS); toxic elements (AA); and the following anions: Cl^- (colorimetric), $SO_4^=$ (titration), $SO_3^=$ (colorimetric), NO_2^- (colorimetric), and NO_3^- (colorimetric). In the second method, a sample bed was prepared in a 50-ml buret column, through which water was pumped up at 1.2 ml/min. Three successive 8-ml portions were removed from the top of the bed, but only the first portion was analyzed. Leachates prepared by the second method were analyzed by the same procedures in the first method.

LIQUIDS AND SLURRIES

No liquids or slurries were sampled.

SOLIDS

Limestone sorbent was grab sampled and riffled to a 1-quart sample, but since very little limestone was used with the low sulfur coal in this study, limestone was not analyzed. Bed discard, cyclone 1 discard, and cyclone 2 discard were sampled hourly and composited. These three samples were analyzed for: particle size distribution by sieving and weighing; anions (Cl^-, F^-, $SO_4^=$, $SO_3^=$, NO_2^-, NO_3^-) by various colorimetric or titrimetric methods; trace metals by SSMS; toxic elements (Hg, As, Se, Sb) by AA; particle morphology by scanning electron microscope; major elements (Al, Ca, Fe, K, Na, Si) by optical emission spectroscopy; and organic classes by LC-IR. The anthracite fuel feed was grab sampled from the feed hopper and analyzed as follows: trace metals by SSMS; fuel analyses (moisture, volatile matter, ash, fixed carbon, C, H, N, S, O, Btu, and sulfur) by ASTM methods; organic classes by LC-IR; particle size distribution by sieving and weighing; toxic elements (Hg, As, Se, Sb) by AA; major elements (Al, Ca, Fe, K, Na, Si) by optical emission spectroscopy; biological analyses by Ames' test, prescreen confluency assay and bacterial toxicity; CO_2^-, Cl^- and F^- by ASTM methods.

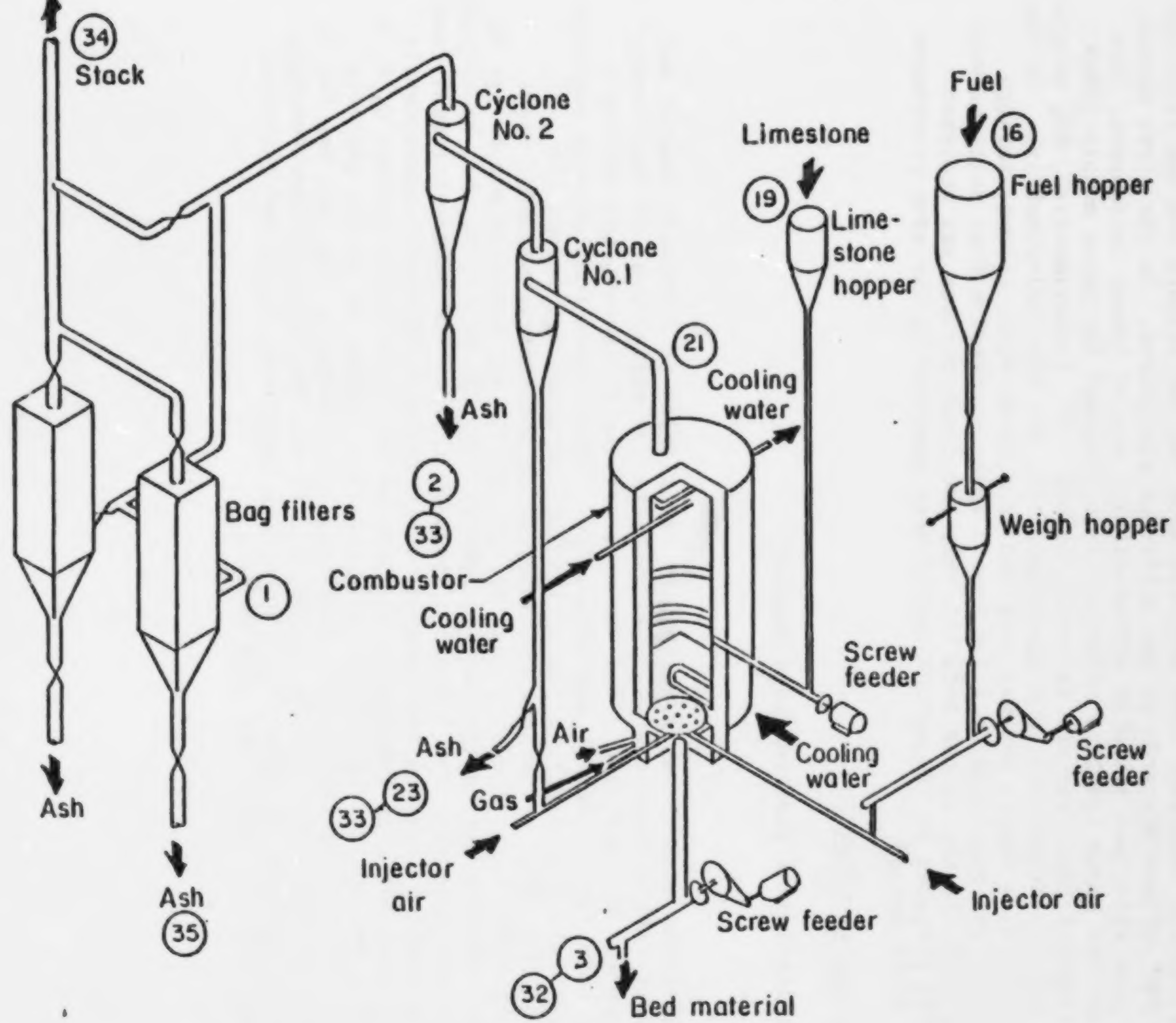
34
Stack
Cyclone No. 2
Fuel
16
Limestone
19
Lime-stone hopper
Fuel hopper
Cyclone No.1
21
Cooling water
Ash
2
33
Bag filters
Weigh hopper
Combustor
1
Cooling water
Screw feeder
Ash
Air
Cooling water
Screw feeder
Ash
33
23
Gas
Injector air
Ash
35
Screw feeder
Injector air
3
32
Bed material

Figure 1. MERC atmospheric fluidized-bed combustor.

TABLE 1. SPARK SOURCE MASS SPECTROSCOPY
COAL CULM FROM FEED HOPPER
(ppmw)

	Run				Run		
	2	3	4		2	3	4
U	1	1	1	Pd	<0.5	<0.3	<2
Th	10	2	10	Rh	<0.5	<0.5	<0.5
Bi	<0.1	0.1	<0.2	Ru	<0.3	<1	<0.3
Pb	3	3	3	Mo	5	2	5
Tl	0.2	0.2	0.2	Nb	10	10	20
Hg	<0.3	<0.3	<0.3	Zr	200	50	200
Au	<0.1	<0.2	<0.1	Y	30	10	50
Pt	<0.3	<0.5	<0.5	Sr	100	100	200
Ir	<0.2	<0.3	<0.2	Rb	200	200	100
Os	<0.2	<0.3	<0.2	Br	0.3	0.5	1
Re	<0.2	<0.3	<0.2	Se	≤20	≤5	≤20
W	<0.5	<2	<2	As	20	10	10
Ta	<0.5	<0.5	<1	Ge	1	5	3
Hf	2	3	3	Ga	20	5	5
Lu	0.2	0.2	0.2	Zn	5	1	5
Yb	1	3	5	Cu	10	5	5
Tm	0.3	1	1	Ni	50	50	100
Er	2	2	2	Co	2	2	10
Ho	1	1	1	Fe	~1%	5,000	~1%
Dy	10	5	10	Mn	30	20	10
Tb	<0.5	0.5	<1	Cr	50	30	100
Gd	2	3	5	V	200	100	100
Eu	2	2	2	Ti	3,000	2,000	3,000
Sm	10	10	10	Sc	10	10	50
Nd	50	30	50	Ca	300	300	200
Pr	10	5	20	K	~3%	~2%	~1%
Ce	100	50	100	Cl	5	5	0.5
La	50	20	50	S	1,000	100	100
Ba	2,000	1,000	2,000	P	100	30	100
Cs	50	50	10	Si	~15%	~15%	~15%
I	0.1	0.1	0.05	Al	~15%	~15%	~15%
Te	<0.5	<0.3	<0.3	Mg	2,000	2,000	1,000
Sb	1	1	2	Na	2,000	500	300
Sn	3	3	5	F	50	20	2
In	<2	<2	<2	B	20	50	20
Cd	5	0.3	1	Be	0.5	0.5	1
Ag	0.3	0.1	0.2	Li	10	10	20

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY
NBS COAL--SRM 1633
(ppmw)

U	2	Dy	10	Rh	<0.5	Cr	10
Th	10	Tb	2	Ru	<0.5	V	100
Bi	0.5	Gd	10	Mo	20	Ti	3,000
Pb	30	Eu	2	Nb	5	Sc	20
Tl	1	Sm	20	Zr	100	Ca	~5%
Hg	<0.3	Nd	30	Y	200	K	~2%
Au	<0.1	Pr	10	Sr	2,000	Cl	10
Pt	<0.3	Ce	100	Rb	300	S	300
Ir	<0.2	La	30	Br	1	P	200
Os	<0.2	Ba	3,000	Se	≤10	Si	~15%
Re	<0.1	Cs	10	As	30	Al	~15%
W	<0.5	I	0.3	Ge	20	Mg	~1%
Ta	<0.5	Te	<0.5	Ga	3	Na	2,000
Hf	1	Sb	1	Zn	100	F	3
Lu	0.3	Sn	5	Cu	100	B	50
Yb	3	In	<3	Ni	100	Be	5
Tm	1	Cd	<0.5	Co	30	Li	50
Er	3	Ag	1	Fe	~1%		
Ho	1	Pd	<2	Mn	200		

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
CYCLONE NUMBER 1--RUN 2*
(>4.6 μ particles [ppmw])

U	10	Dy	50	Rh	<10	Cr	500
Th	100	Tb	10	Ru	<0.5	V	300
Bi	20	Gd	100	Mo	50	Ti	~2%
Pb	300	Eu	20	Nb	100	Sc	200
Tl	5	Sm	100	Zr	500	Ca	3,000
Hg	<0.5	Nd	500	Y	200	K	~2%
Au	<0.1	Pr	100	Sr	300	Cl	20
Pt	<0.3	Ce	500	Rb	1,000	S	3,000
Ir	<0.2	La	300	Br	100	P	500
Os	<0.2	Ba	2,000	Se	≤500†	Si	~30%
Re	<0.2	Cs	100	As	1,000	Al	~10%
W	3	I	10	Ge	50	Mg	~1%
Ta	<5	Te	2	Ga	200	Na	3,000
Hf	20	Sb	50	Zn	20	F	500
Lu	5	Sn	100	Cu	300	B	5
Yb	30	In	<1†	Ni	200	Be	0.2
Tm	5	Cd	5	Co	30	Li	20
Er	50	Ag	3	Fe	~3%		
Ho	20	Pd	≤20†	Mn	100		

*Collection by modified method 5 train located at bag collector inlet--stream 1.

†Memory from previous sample.

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
CYCLONE NUMBER 1--RUN 3*
(>4.6 μ particles [ppmw])

U	5	Dy	10	Rh	<10	Cr	300
Th	50	Tb	5	Ru	<0.5	V	500
Bi	10	Gd	50	Mo	20	Ti	~2%
Pb	200	Eu	10	Nb	100	Sc	200
Tl	2	Sm	20	Zr	500	Ca	1,000
Hg	<0.5	Nd	200	Y	100	K	5,000
Au	<0.1	Pr	50	Sr	200	Cl	20
Pt	<0.3	Ce	100	Rb	300	S	1,000
Ir	<0.2	La	200	Br	30	P	500
Os	<0.2	Ba	500	Se	≤200†	Si	~30%
Re	<0.2	Cs	10	As	200	Al	~10%
W	0.3	I	1	Ge	20	Mg	5,000
Ta	<1	Te	0.5	Ga	200	Na	1,000
Hf	<0.5	Sb	10	Zn	20	F	200
Lu	<0.2	Sn	10	Cu	300	B	5
Yb	<0.5	In	<1†	Ni	100	Be	0.3
Tm	3	Cd	1	Co	30	Li	20
Er	20	Ag	1	Fe	~3%		
Ho	5	Pd	≤3†	Mn	50		

*Collection by modified method 5 train located at bag collector inlet--stream 1.

†Memory from previous sample.

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY
CYCLONE NUMBER 1--RUN 4*
(>4.6 μ particles [ppmw])

U	10	Dy	10	Rh	<10	Cr	500
Th	50	Tb	1	Ru	<0.5	V	300
Bi	10	Gd	20	Mo	20	Ti	~1%
Pb	500	Eu	5	Nb	20	Sc	30
Tl	3	Sm	10	Zr	500	Ca	2,000
Hg	<0.5	Nd	200	Y	100	K	~1%
Au	<0.1	Pr	20	Sr	200	Cl	20
Pt	<0.3	Ce	100	Rb	300	S	1,000
Ir	<0.2	La	50	Br	10	P	100
Os	<0.2	Ba	500	Se	≤500	Si	~30%
Re	<0.2	Cs	30	As	50	Al	~10%
W	3	I	2	Ge	20	Mg	5,000
Ta	<2	Te	<0.5	Ga	30	Na	2,000
Hf	<0.3	Sb	10	Zn	100	F	100
Lu	<0.3	Sn	10	Cu	100	B	10
Yb	<0.5	In	<1†	Ni	100	Be	0.3
Tm	2	Cd	3	Co	30	Li	50
Er	10	Ag	1	Fe	~5%		
Ho	1	Pd	≤3†	Mn	100		

*Collection by modified method 5 train located at bag collector inlet--stream 1.

†Memory from previous sample.

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY CYCLONE NUMBER 2 AND FILTER--RUN 2*

(<4.6 μ [ppmw])

U	5	Dy	10	Rh	<3	Cr	300
Th	10	Tb	2	Ru	<0.5	V	300
Bi	10	Gd	20	Mo	20	Ti	~2%
Pb	100	Eu	10	Nb	50	Sc	200
Tl	1	Sm	30	Zr	500	Ca	2,000
Hg	<0.5	Nd	100	Y	100	K	5,000
Au	<0.1	Pr	30	Sr	200	Cl	30
Pt	<0.3	Ce	200	Rb	200	S	3,000
Ir	<0.1	La	100	Br	10	P	500
Os	<0.2	Ba	1,000	Se	≤100†	Si	~30%
Re	<0.2	Cs	50	As	300	Al	~10%
W	1	I	0.3	Ge	50	Mg	~1%
Ta	<1	Te	1	Ga	100	Na	3,000
Hf	3	Sb	10	Zn	50	F	1,000
Lu	1	Sn	30	Cu	100	B	20
Yb	10	In	<1†	Ni	200	Be	0.3
Tm	1	Cd	3	Co	50	Li	10
Er	10	Ag	1	Fe	~5%		
Ho	2	Pd	≤3†	Mn	200		

*Collection by modified method 5 train located at bag collector inlet--stream 1.

†Memory from previous sample.

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY CYCLONE NUMBER 2 AND FILTER--RUN 3*

(<4.6 μ [ppmw])

U	5	Dy	30	Rh	<3	Cr	300
Th	20	Tb	3	Ru	<0.5	V	1,000
Bi	5	Gd	50	Mo	30	Ti	~2%
Pb	100	Eu	10	Nb	100	Sc	200
Tl	1	Sm	50	Zr	500	Ca	3,000
Hg	<0.5	Nd	300	Y	200	K	~1%
Au	<0.1	Pr	50	Sr	300	Cl	30
Pt	<0.3	Ce	500	Rb	300	S	5,000
Ir	<0.1	La	200	Br	10	P	1,000
Os	<0.2	Ba	2,000	Se	100†	Si	~30%
Re	<0.2	Cs	50	As	200	Al	~10%
W	1	I	1	Ge	100	Mg	~1%
Ta	<1	Te	2	Ga	100	Na	3,000
Hf	5	Sb	10	Zn	100	F	1,000
Lu	1	Sn	30	Cu	100	B	20
Yb	10	In	<1†	Ni	200	Be	1
Tm	2	Cd	3	Co	50	Li	20
Er	10	Ag	1	Fe	~10%		
Ho	5	Pd	≤10†	Mn	200		

*Collection by modified method 5 train located at bag collector inlet--stream 1.

†Memory from previous sample.

TABLE 8. SPARK SOURCE MASS SPECTROSCOPY CYCLONE NUMBER 2 AND FILTER--RUN 4*
(<4.6 μ [ppmw])

U	10	Dy	30	Rh	<2	Cr	300
Th	20	Tb	3	Ru	<0.5	V	500
Bi	10	Gd	50	Mo	30	Ti	~3%
Pb	100	Eu	10	Nb	100	Sc	300
Tl	1	Sm	50	Zr	500	Ca	2,000
Hg	<0.5	Nd	300	Y	200	K	5,000
Au	<0.1	Pr	50	Sr	200	Cl	30
Pt	<0.3	Ce	200	Rb	200	S	5,000
Ir	<0.1	La	200	Br	10	P	1,000
Os	<0.2	Ba	2,000	Se	≤200†	Si	~30%
Re	<0.3	Cs	50	As	300	Al	~10%
W	3	I	2	Ge	100	Mg	~1%
Ta	<2	Te	1	Ga	100	Na	3,000
Hf	10	Sb	10	Zn	50	F	1,000
Lu	1	Sn	30	Cu	100	B	20
Yb	10	In	<1†	Ni	200	Be	0.3
Tm	2	Cd	3	Co	50	Li	20
Er	20	Ag	1	Fe	~10%		
Ho	5	Pd	≤10†	Mn	200		

*Collection by modified method 5 train located at bag collector inlet--stream 1.

†Memory from previous sample.

TABLE 9. SPARK SOURCE MASS SPECTROSCOPY
BED REJECT MATERIAL
(ppmw)

	Run				Run		
	2	3	4		2	3	4
U	2	2	2	Pd	<2	<2	<3
Th	10	10	10	Rh	<0.5	<0.5	<0.5
Bi	<1	0.3	<1	Ru	<0.5	<0.5	<0.5
Pb	100	100	100	Mo	2	2	2
Tl	2	1	2	Nb	10	10	20
Hg	<0.3	<0.3	<0.3	Zr	100	50	100
Au	<0.1	<0.1	<0.1	Y	50	30	30
Pt	<0.3	<0.3	<0.3	Sr	300	100	100
Ir	<0.2	<0.2	<0.2	Rb	1,000	1,000	1,000
Os	<0.2	<0.2	<0.2	Br	1	0.5	0.5
Re	<0.2	<0.2	<0.2	Se	≤5	≤2	≤1
W	<1	<1	<1	As	20	20	20
Ta	<0.5	<0.5	<0.5	Ge	5	2	2
Hf	2	2	3	Ga	20	20	30
Lu	0.5	0.5	0.5	Zn	50	20	20
Yb	3	3	3	Cu	50	10	30
Tm	0.5	0.5	0.5	Ni	20	20	10
Er	5	3	5	Co	5	5	3
Ho	2	2	2	Fe	5,000	5,000	5,000
Dy	10	3	10	Mn	30	30	20
Tb	3	1	2	Cr	50	10	20
Gd	5	3	3	V	100	100	100
Eu	5	3	5	Ti	3,000	3,000	5,000
Sm	10	10	5	Sc	10	10	10
Nd	50	50	50	Ca	5,000	~1%	~1%
Pr	20	5	10	K	~10%	~10%	~10%
Ce	200	100	100	Cl	20	10	10
La	50	30	50	S	500	1,000	300
Ba	3,000	500	1,000	P	100	50	200
Cs	100	30	50	Si	~15%	~15%	~15%
I	0.05	0.05	<0.05	Al	~15%	~15%	~15%
Te	<0.3	<0.5	<0.3	Mg	~1%	5,000	~1%
Sb	1	1	1	Na	3,000	2,000	2,000
Sn	10	5	5	F	50	30	30
In	<1	<1	<3	B	20	50	100
Cd	<0.5	<0.5	<0.5	Be	0.5	1	2
Ag	2	0.2	0.3	Li	30	50	-300

TABLE 10. SPARK SOURCE MASS SPECTROSCOPY FBC CYCLONE NUMBER 1 PARTICULATES--RUN 3
(ppmw)

U	1	Dy	2	Rh	<0.5	Cr	50
Th	10	Tb	0.3	Ru	<0.5	V	100
Bi	<0.1	Gd	3	Mo	3	Ti	3,000
Pb	30	Eu	2	Nb	20	Sc	20
Tl	0.5	Sm	10	Zr	200	Ca	5,000
Hg	<0.3	Nd	30	Y	20	K	~5%
Au	<0.1	Pr	10	Sr	100	Cl	2
Pt	<0.3	Ce	100	Rb	200	S	100
Ir	<0.2	La	50	Br	0.5	P	100
Os	<0.2	Ba	2,000	Se	≤5	Si	~15%
Re	<0.2	Cs	50	As	30	Al	~15%
W	<1	I	0.05	Ge	5	Mg	2,000
Ta	<0.2	Te	<0.3	Ga	20	Na	1,000
Hf	0.5	Sb	2	Zn	10	F	10
Lu	0.2	Sn	10	Cu	50	B	10
Yb	2	In	<1	Ni	30	Be	0.2
Tm	0.3	Cd	<0.5	Co	10	Li	30
Er	2	Ag	3	Fe	~1%		
Ho	0.3	Pd	<1	Mn	30		

TABLE 11. SPARK SOURCE MASS SPECTROSCOPY FBC CYCLONE NUMBER 1 PARTICULATES--RUN 4
(ppmw)

U	2	Dy	5	Rh	<0.5	Cr	30
Th	10	Tb	1	Ru	<0.5	V	50
Bi	<0.2	Gd	3	Mo	2	Ti	3,000
Pb	30	Eu	2	Nb	20	Sc	20
Tl	2	Sm	5	Zr	100	Ca	5,000
Hg	<0.3	Nd	30	Y	10	K	~5%
Au	<0.1	Pr	10	Sr	100	Cl	5
Pt	<0.3	Ce	50	Rb	1,000	S	300
Ir	<0.2	La	30	Br	0.5	P	50
Os	<0.2	Ba	1,000	Se	≤5	Si	~15%
Re	<0.2	Cs	30	As	10	Al	~15%
W	<1	I	0.1	Ge	3	Mg	~1%
Ta	<1	Te	<0.3	Ga	20	Na	1,000
Hf	0.5	Sb	1	Zn	10	F	20
Lu	0.1	Sn	10	Cu	20	B	20
Yb	2	In	<10	Ni	30	Be	0.5
Tm	0.3	Cd	<0.5	Co	5	Li	30
Er	2	Ag	0.3	Fe	5,000		
Ho	1	Pd	<1	Mn	30		

TABLE 12. SPARK SOURCE MASS SPECTROSCOPY FBC CYCLONE NUMBER 2 PARTICULATES (ppmw)

	Run				Run		
	2	3	4		2	3	4
U	2	2	1	Pd	<2	<1	<2
Th	20	10	10	Rh	<0.5	<0.5	<0.5
Bi	1	1	1	Ru	<0.3	<0.3	<0.5
Pb	100	200	30	Mo	3	3	3
Tl	0.5	2	0.5	Nb	20	10	10
Hg	<0.3	<0.3	<0.3	Zr	200	100	100
Au	<0.1	<0.1	<0.1	Y	50	20	30
Pt	<0.3	<0.3	<0.3	Sr	100	100	300
Ir	<0.2	<0.2	<0.2	Rb	500	500	500
Os	<0.2	<0.2	<0.2	Br	0.5	0.5	1
Re	<0.2	<0.2	<0.2	Se	≤20	≤10	≤10
W	<1	<1	<0.5	As	30	50	20
Ta	<1	<1	<0.5	Ge	5	5	2
Hf	2	2	1	Ga	20	20	3
Lu	0.1	0.1	0.1	Zn	30	30	100
Yb	2	2	3	Cu	30	30	30
Tm	0.2	0.1	0.5	Ni	50	50	50
Er	2	2	2	Co	10	10	10
Ho	1	1	0.5	Fe	~1%	~1%	~1%
Dy	5	5	3	Mn	50	30	20
Tb	1	1	1	Cr	30	30	20
Gd	5	5	3	V	200	200	50
Eu	2	2	2	Ti	3,000	3,000	3,000
Sm	10	20	5	Sc	50	50	20
Nd	100	30	30	Ca	3,000	3,000	5,000
Pr	20	10	5	K	~3%	~5%	~5%
Ce	100	100	50	Cl	2	2	2
La	100	50	50	S	300	500	50
Ba	2,000	2,000	500	P	100	100	100
Cs	30	50	30	Si	~15%	~15%	~15%
I	<0.05	<0.05	0.3	Al	~15%	~15%	~15%
Te	<0.3	<0.3	<0.5	Mg	~1%	~1%	~1%
Sb	2	3	1	Na	2,000	2,000	3,000
Sn	5	10	5	F	20	30	50
In	<5	<20	<3	B	30	30	30
Cd	<0.5	<0.3	<0.3	Be	20	0.5	0.5
Ag	0.1	0.2	1	Li	50	30	30

TABLE 13. SPARK SOURCE MASS SPECTROSCOPY
LEACHATE OF BED REJECT MATERIAL--RUN 3 (ULTRASONIC SHAKING)*
(ng/ml)

	Shake Number			Shake Number	
	1	10		1	10
U	†	†	Pd	<0.1	<0.4
Th	†	†	Rh	<0.2	<0.2
Bi	<0.2	<0.4	Ru	<0.3	<0.4
Pb	2	<1	Mo	20	4
Tl	<0.2	<1	Nb	<0.05	<0.15
Hg	<0.5	<0.4	Zr	<0.2	<2
Au	<1	<0.4	Y	0.5	<0.15
Pt	<0.3	<1	Sr	100	1
Ir	<0.2	<1	Rb	20	<0.15
Os	†	†	Br	20	4
Re	<0.3	<0.4	Se	<0.7	<0.3
W	0.5	<1	As	10	0.7
Ta	<20	<4	Ge	2	<0.4
Hf	<1	<3	Ga	0.3	<0.2
Lu	<0.1	<0.2	Zn	6	15
Yb	<0.3	<1	Cu	3	4
Tm	<0.1	<0.2	Ni	20	20
Er	†	†	Co	<0.2	0.2
Ho	<0.1	<0.2	Fe	40	80
Dy	<0.3	<1	Mn	0.3	2
Tb	<0.1	<0.2	Cr	10	20
Gd	<0.3	<1	V	20	2
Eu	<0.1	<0.4	Ti	40	10
Sm	<0.5	<1	Sc	<0.06	<0.15
Nd	<0.3	<1	Ca	100,000	700
Pr	<0.1	<0.2	K	15,000	400
Ce	<0.1	<0.2	Cl	300	200
La	<0.1	<0.2	S	10,000	200
Ba	150	0.3	P	3	10
Cs	0.5	<0.2	Si	2,000	70
I	2	0.3	Al	150	60
Te	<0.2	<1	Mg	20	300
Sb	<0.1	<0.4	Na	1,500	600
Sn	<0.2	<1	F	<1	<1
In	<0.2	<0.2	B	40	0.7
Cd	1.5	<3	Be	<0.006	<0.01
Ag	<0.5	<0.3	Li	30	0.7

*See leachate preparation in summary.
†Not reported.

TABLE 14. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF BED REJECT MATERIAL RUN 3 (COLUMN)*

(ng/ml)

U	†	Dy	<0.2	Rh	<0.15	Cr	7
Th	†	Tb	<0.07	Ru	<0.15	V	1,500
Bi	0.15	Gd	<0.2	Mo	300	Ti	400
Pb	1.5	Eu	<0.07	Nb	<0.15	Sc	<0.04
Tl	0.15	Sm	<0.2	Zr	<0.15	Ca	60,000
Hg	<0.4	Nd	<0.2	Y	<0.15	K	100,000
Au	<0.7	Pr	<0.07	Sr	150	Cl	15,000
Pt	<0.2	Ce	<0.07	Rb	300	S	30,000
Ir	0.15	La	<0.07	Br	50	P	15
Os	†	Ba	15	Se	≤3	Si	3,000
Re	<0.2	Cs	3	As	300	Al	400
W	1.5	I	0.5	Ge	3	Mg	700
Ta	<40	Te	0.2	Ga	5	Na	1,000
Hf	<0.6	Sb	15	Zn	15	F	4
Lu	<0.07	Sn	<0.15	Cu	7	B	150
Yb	<0.2	In	<0.7	Ni	30	Be	<0.004
Tm	<0.07	Cd	4	Co	<0.15	Li	100
Er	†	Ag	0.5	Fe	70		
Ho	0.07	Pd	<0.15	Mn	0.7		

*See leachate preparation in summary.
†Not reported.

TABLE 15. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF BED REJECT MATERIAL RUN 4 (ULTRASONIC SHAKING)* (ng/ml)

	Shake Number			Shake Number	
	1	10		1	10
U	†	†	Pd	<0.4	<0.2
Th	†	†	Rh	<0.2	<0.05
Bi	<0.4	<0.1	Ru	<0.7	<0.2
Pb	<1.5	<0.4	Mo	1	0.7
Tl	<1	<0.4	Nb	<0.1	<0.05
Hg	<1	<0.4	Zr	<3	<0.1
Au	<1	<0.4	Y	<0.2	<0.05
Pt	<1	<0.4	Sr	1	0.5
Ir	<1	<0.4	Rb	7	1.5
Os	†	<0.4	Br	4	2
Re	<1	<0.4	Se	<0.7	<1
W	<1	<2	As	7	1
Ta	<10	<1	Ge	<0.04	<0.3
Hf	<2	<0.1	Ga	2	0.2
Lu	<0.2	<0.4	Zn	7	300
Yb	<1	<0.4	Cu	40	200
Tm	<0.2		Ni	2	10
Er	†		Co	0.2	0.15
Ho	<0.2	<0.1	Fe	2,000	600
Dy	<1	<0.4	Mn	40	0.3
Tb	<0.2	<0.1	Cr	4	2
Gd	<1	<0.4	V	5	1.5
Eu	<0.4	<0.2	Ti	700	50
Sm	<1	<0.2	Sc	0.3	0.6
Nd	1.5	<0.3	Ca	50	70
Pr	0.2	<0.1	K	50,000	9,000
Ce	1	0.2	Cl	402	150
La	0.5	0.2	S	72	70
Ba	15	1	P	4,000	100
Cs	0.5	0.1	Si	100,000	15,000
I	<0.2	0.06	Al	70,000	1,500
Te	<1	<0.3	Mg	1,500	150
Sb	<0.4	<0.2	Na	1,000	500
Sn	<0.7	<0.3	F	<0.7	<0.3
In	<0.3	<206	B	10	30
Cd	<2	<1.5	Be	0.07	0.03
Ag	<0.3	<0.1	Li	70	3

*See leachate preparation in summary.
†Not reported.

TABLE 16. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF BED REJECT MATERIAL RUN 4 (COLUMN)*

(ng/ml)

U	†	Dy	<1.5	Rh	<0.2	Cr	<0.3
Th	†	Tb	<0.5	Ru	<0.6	V	600
Bi	<0.5	Gd	4.5	Mo	2	Ti	60
Pb	<2	Eu	<1	Nb	<0.2	Sc	<0.2
Tl	<1.5	Sm	<1	Zr	<1	Ca	200
Hg	<1.5	Nd	<1.5	Y	<0.2	K	6,000
Au	<2	Pr	<0.5	Sr	0.4	Cl	70
Pt	<1.5	Ce	<0.5	Rb	3	S	40,000
Ir	<1.5	La	<0.4	Br	<4	P	6
Os	†	Ba	0.4	Se	<0.3	Si	6,000
Re	<1.5	Cs	<0.3	As	150	Al	2,000
W	<1.5	I	<0.3	Ge	<2	Mg	1,500
Ta	<10	Te	<1.5	Ga	<0.3	Na	40,000
Hf	<4	Sb	<0.7	Zn	1.5	F	<1.5
Lu	<0.5	Sn	<1	Cu	0.6	B	60
Yb	<1.5	In	<0.3	Ni	4	Be	0.15
Tm	<1.5	Cd	<7	Co	0.2	Li	10,000
Er	†	Ag	<0.7	Fe	400		
Ho	<0.5	Pd	<1	Mn	10		

*See leachate preparation in summary.
†Not reported.

TABLE 17. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF PFBC CYCLONE NUMBER 1 PARTICULATES RUN 4 (ULTRASONIC SHAKE)*

(ng/ml)

	Shake Number			Shake Number	
	1	10		1	10
U	†	†	Pd	<0.01	<0.06
Th	†	†	Rh	<0.03	<0.03
Bi	<0.1	<0.06	Ru	<0.1	<0.06
Pb	<0.4	0.1	Mo	0.3	0.2
Tl	<0.2	<0.2	Nb	<0.03	<0.02
Hg	<0.2	<0.2	Zr	<0.1	<0.15
Au	<0.5	<0.6	Y	<0.03	<0.02
Pt	<0.2	<0.2	Sr	3	0.06
Ir	<0.2	<0.2	Rb	2	1
Os	†	†	Br	1	<1
Re	<0.2	<0.2	Se	0.5	<0.06
W	<0.2	<0.2	As	5	1
Ta	<3	<3	Ge	<0.1	<0.01
Hf	<0.6	<0.4	Ga	<0.05	0.1
Lu	<0.05	<0.03	Zn	4	6
Yb	<0.2	<0.2	Cu	2	6
Tm	<0.05	<0.03	Ni	3	3
Er	†	†	Co	0.5	0.04
Ho	<0.05	<0.3	Fe	20	100
Dy	<0.2	<0.2	Mn	3	0.6
Tb	<0.05	<0.03	Cr	0.5	0.3
Gd	<0.2	<0.2	V	1.5	1
Eu	<0.1	<0.06	Ti	10	20
Sm	<0.2	<0.2	Sc	<0.03	<0.02
Nd	<0.2	<0.2	Ca	2,200	5
Pr	<0.05	<0.02	K	10,000	50
Ce	<0.05	<0.04	Cl	15	30
La	<0.05	0.1	S	1,000	70
Ba	1	0.6	P	3	4
Cs	<0.05	0.04	Si	200	400
I	<0.05	<0.02	Al	5	600
Te	<0.2	<0.1	Mg	200	40
Sb	<0.1	<0.04	Na	1,500	10
Sn	<0.2	<0.1	F	0.4	<0.2
In	<0.05	<0.02	B	0.3	0.3
Cd	<0.6	<0.2	Be	0.01	0.02
Ag	<0.07	<0.03	Li	200	0.4

*See leachate preparation in summary.
†Not reported.

TABLE 18. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF PFBC CYLONE NUMBER 1 PARTICULATES RUN 4 (COLUMN)* (ng/ml)

U	†	Dy	3	Rh	<1	Cr	150
Th	†	Tb	1	Ru	<0.2	V	100
Bi	<0.4	Gd	6	Mo	15	Ti	60
Pb	15	Eu	1.5	Nb	<0.1	Sc	1
Tl	1.5	Sm	6	Zr	<1.5	Ca	70,000
Hg	<1	Nd	15	Y	60	K	300,000
Au	≤6	Pr	3	Sr	1,500	Cl	300
Pt	<0.7	Ce	60	Rb	1,000	S	60,000
Ir	<0.4	La	30	Br	60	P	20
Os	†	Ba	50	Se	15	Si	6,000
Re	<0.7	Cs	1.5	As	300	Al	1,500
W	<0.7	I	0.15	Ge	15	Mg	30,000
Ta	<150	Te	0.6	Ga	6	Na	2,000
Hf	<2	Sb	6	Zn	600	F	60
Lu	0.15	Sn	0.6	Cu	100	B	100
Yb	1.5	In	<0.3	Ni	300	Be	0.7
Tm	0.3	Cd	30	Co	150	Li	600
Er	†	Ag	<0.4	Fe	3,000		
Ho	1	Pd	<0.4	Mn	1,500		

*See leachate preparation in summary.
†Not reported.

TABLE 19. SPARK SOURCE MASS SPECTROSCOPY
LEACHATE OF PFBC CYCLONE NUMBER 2 PARTICULATES
RUN 2 (ULTRASONIC SHAKING)*
(ng/ml)

	Shake Number			Shake Number	
	1	10		1	10
U	†	†	Pd	<0.07	<0.07
Th	†	†	Rh	≤0.04	<0.03
Bi	<0.04	<0.07	Ru	<0.2	<0.07
Pb	0.3	0.6	Mo	0.7	0.3
Tl	<0.15	<0.15	Nb	<0.02	<0.02
Hg	<0.15	<0.15	Zr	0.3	<0.1
Au	<0.15	<1	Y	<0.02	<0.02
Pt	<0.15	<0.15	Sr	7	0.15
Ir	<0.07	<0.15	Rb	0.7	1.5
Os	<0.15	<0.15	Br	1.5	5
Re	<0.07	<0.07	Se	1.5	<0.07
W	<0.15	<0.15	As	2	4
Ta	<3	<10	Ge	<0.3	<0.01
Hf	<0.5	<0.5	Ga	<0.4	<0.1
Lu	<0.04	<0.04	Zn	0.7	10
Yb	<0.15	<0.15	Cu	0.4	15
Tm	<0.04	<0.04	Ni	0.7	6
Er	<0.15	<0.15	Co	0.2	0.15
Ho	<0.04	<0.04	Fe	30	400
Dy	<0.15	<0.15	Mn	0.7	0.7
Tb	<0.07	<0.04	Cr	0.07	0.4
Gd	<0.2	<0.15	V	1.5	2
Eu	≤0.07	<0.07	Ti	4	30
Sm	<0.15	<0.15	Sc	<0.07	<0.02
Nd	<0.15	<0.15	Ca	400	150
Pr	<0.04	<0.03	K	1,000	1,000
Ce	<0.04	<0.03	Cl	15	100
La	<0.04	0.2	S	2	4,000
Ba	0.7	0.7	P	2	20
Cs	0.04	0.1	Si	7	1,500
I	0.15	<0.03	Al	70	700
Te	<0.2	<0.1	Mg	70	150
Sb	†	†	Na	300	150
Sn	<0.07	<0.1	F	0.7	<0.15
In	<0.4	<0.03	B	1.5	4
Cd	<0.5	<0.03	Be	0.04	0.01
Ag	<0.04	<0.04	Li	5	30

*See leachate preparation in summary.
†Not reported.

TABLE 20. SPARK SOURCE MASS SPECTROSCOPY
LEACHATE OF PFBC CYCLONE NUMBER 2 PARTICULATES
RUN 2 (COLUMN)*
(ng/ml)

U	†	Dy	2	Rh	<1.5	Cr	200
Th	†	Tb	0.7	Ru	<2	V	70
Bi	<0.5	Gd	4	Mo	30	Ti	150
Pb	15	Eu	1.5	Nb	<0.15	Sc	0.4
Tl	4	Sm	4	Zr	<1	Ca	300,000
Hg	<1.5	Nd	20	Y	20	K	500,000
Au	≤10	Pr	4	Sr	2,000	Cl	200
Pt	<1	Ce	40	Rb	1,000	S	200,000
Ir	<0.5	La	20	Br	100	P	30
Os	<1	Ba	40	Se	70	Si	20,000
Re	<0.5	Cs	20	As	400	Al	20,000
W	<1	I	0.4	Ge	40	Mg	100,000
Ta	<200	Te	1.5	Ga	15	Na	7,000
Hf	<3	Sb	†	Zn	1,500	F	150
Lu	<0.2	Sn	0.4	Cu	150	B	700
Yb	<1.5	In	≤0.4	Ni	1,500	Be	0.7
Tm	<0.2	Cd	30	Co	1,500	Li	1,500
Er	0.7	Ag	<0.5	Fe	4,000		
Ho	0.4	Pd	<0.5	Mn	4,000		

*See leachate preparation in summary.
†Not reported.

TABLE 21. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF PFBC CYCLONE NUMBER 2 PARTICULATES RUN 3 (ULTRASONIC SHAKING)*
(ng/ml)

	Shake Number			Shake Number	
	1	10		1	10
U	†	†	Pd	<0.3	<0.5
Th	†	†	Rh	<0.1	<0.3
Bi	<0.06	<0.06	Ru	<0.3	<0.5
Pb	0.4	<2	Mo	40	5
Tl	<0.1	<1	Nb	<0.03	<0.15
Hg	<0.2	<1	Zr	<0.4	<2
Au	<0.6	<3	Y	0.03	<0.15
Pt	<0.2	<1	Sr	500	0.5
Ir	<0.1	<1	Rb	50	1
Os	<0.2	<1	Br	20	2
Re	<0.1	<0.5	Se	20	<0.4
W	<0.2	<1	As	20	1.5
Ta	≤40	<15	Ge	<3	<0.5
Hf	<0.7	<3	Ga	0.3	<0.3
Lu	<0.05	<0.3	Zn	20	4
Yb	<0.2	<1	Cu	5	5
Tm	<0.05	<0.3	Ni	20	<1.5
Er	<0.2	<1	Co	<1	<0.15
Ho	<0.05	<0.3	Fe	100	400
Dy	<0.2	<1	Mn	20	0.7
Tb	<0.05	<0.3	Cr	10	1
Gd	<0.2	<1	V	30	3
Eu	<0.1	<0.5	Ti	20	40
Sm	<0.2	<1	Sc	<0.1	<0.15
Nd	<0.2	<1	Ca	100,000	300
Pr	<0.05	<0.3	K	40,000	400
Ce	0.05	<0.3	Cl	200	70
La	<0.05	0.1	S	150,000	30
Ba	10	1	P	30	15
Cs	0.5	<0.3	Si	500	100
I	0.5	<0.3	Al	100	1,500
Te	<0.2	<1	Mg	5,000	100
Sb	†	†	Na	4,000	60
Sn	0.5	<1	F	2	<1
In	<0.05	<0.3	B	30	1
Cd	<0.6	<3	Be	0.1	<0.01
Ag	0.05	<0.4	Li	300	70

*See leachate preparation in summary.
†Not reported.

TABLE 22. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF PFBC CYCLONE NUMBER 2 PARTICULATES

RUN 3 (COLUMN)*

(ng/ml)

U	†	Dy	1.5	Rh	<0.6	Cr	6
Th	†	Tb	0.3	Ru	<1.5	V	100
Bi	<0.2	Gd	3	Mo	200	Ti	100
Pb	5	Eu	0.3	Nb	<0.2	Sc	0.5
Tl	0.4	Sm	1	Zr	≤1	Ca	150,000
Hg	<0.4	Nd	4	Y	6	K	150,000
Au	<10	Pr	1	Sr	1,000	Cl	300
Pt	<0.4	Ce	6	Rb	400	S	300,000
Ir	<0.2	La	3	Br	100	P	15
Os	<0.4	Ba	40	Se	40	Si	3,000
Re	<0.2	Cs	15	As	40	Al	3,000
W	<0.4	I	1	Ge	7	Mg	15,000
Ta	<100	Te	0.3	Ga	<0.3	Na	15,000
Hf	<1.5	Sb	†	Zn	200	F	70
Lu	<0.1	Sn	0.4	Cu	60	B	100
Yb	<0.4	In	≤0.4	Ni	40	Be	0.5
Tm	0.1	Cd	5	Co	10	Li	5,000
Er	0.4	Ag	<0.4	Fe	500		
Ho	0.2	Pd	<0.4	Mn	300		

*See leachate preparation in summary.
†Not reported.

TABLE 23. SPARK SOURCE MASS SPECTROSCOPY LEACHATE OF FBC CYCLONE NUMBER 2 PARTICULATES RUN 4 (ULTRASONIC SHAKING)*
(ng/ml)

	Shake Number			Shake Number	
	1	10		1	10
U	†	†	Pd	<0.3	<0.1
Th	†	†	Rh	<0.4	<0.1
Bi	<0.2	<0.05	Ru	<1.5	<0.15
Pb	7	4	Mo	7	1
Tl	0.7	<0.2	Nb	<0.4	0.3
Hg	<0.3	<0.2	Zr	<0.3	7
Au	20	<0.2	Y	30	0.5
Pt	<0.3	<0.2	Sr	700	5
Ir	<0.2	<0.2	Rb	400	20
Os	<0.3	<0.2	Br	50	5
Re	<0.2	<0.1	Se	15	<0.7
W	<0.3	<0.2	As	40	15
Ta	<50	<20	Ge	10	0.7
Hf	<1	<0.6	Ga	<0.2	1.5
Lu	0.07	0.05	Zn	400	60
Yb	1.5	<0.2	Cu	70	15
Tm	0.3	<0.2	Ni	300	10
Er	1.5	<0.2	Co	400	1
Ho	0.7	<0.05	Fe	3,000	15,000
Dy	2	≤0.7	Mn	700	5
Tb	0.7	0.05	Cr	40	10
Gd	7	<0.2	V	40	50
Eu	0.7	0.1	Ti	50	400
Sm	3	0.2	Sc	1.5	1
Nd	20	1.5	Ca	40,000	150
Pr	4	0.3	K	200,000	6,000
Ce	40	1.5	Cl	150	30
La	20	1.5	S	150,000	50
Ba	40	20	P	20	70
Cs	10	1.5	Si	4,000	30,000
I	0.4	0.3	Al	70,000	20,000
Te	0.2	<0.15	Mg	30,000	100
Sb	†	†	Na	7,000	400
Sn	0.4	0.4	F	150	6
In	≤0.3	<0.04	B	40	30
Cd	4	<1	Be	0.7	2.5
Ag	0.5	0.5	Li	3,000	40

*See leachate preparation in summary.
†Not reported.

TABLE 24. SPARK SOURCE MASS SPECTROSCOPY
LEACHATE OF FBC CYCLONE NUMBER 2 PARTICULATES
RUN 4 (COLUMN)*
(ng/ml)

U	†	Dy	15	Rh	<1	Cr	300
Th	†	Tb	5	Ru	<5	V	70
Bi	10	Gd	40	Mo	20	Ti	1,000
Pb	1,000	Eu	7	Nb	3	Sc	10
Tl	3	Sm	50	Zr	20	Ca	150,000
Hg	<1	Nd	100	Y	150	K	150,000
Au	<2	Pr	40	Sr	5,000	Cl	10,000
Pt	<1	Ce	300	Rb	1,000	S	150,000
Ir	<0.5	La	40	Br	3,000	P	1,500
Os	<1	Ba	400	Se	70	Si	30,000
Re	<1	Cs	40	As	200	Al	500,000
W	5	I	7	Ge	40	Mg	300,000
Ta	<50	Te	2	Ga	20	Na	70,000
Hf	3	Sb	†	Zn	5,000	F	1,000
Lu	0.5	Sn	200	Cu	10,000	B	300
Yb	3	In	<3	Ni	500	Be	10
Tm	0.5	Cd	50	Co	300	Li	4,000
Er	10	Ag	10	Fe	700,000		
Ho	3	Pd	<2	Mn	5,000		

*See leachate preparation in summary.
†Not reported.

TABLE 25. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS* FBC LEACHATE SAMPLES (ULTRASONIC SHAKING)† (ppm)

Sample	Shake	Run	Hg	Sb	As
Bed reject material	1	3	<0.005	‡	<0.02
	10	3	<0.005		0.06
	1	4	<0.005		0.44
	10	4	<0.005		0.11
FBC Cyclone #1	1	4	<0.005		0.22
	10	4	<0.005		0.14
FBC Cyclone #2	1	2	<0.005		0.22
	10	2	<0.005		0.30
	1	3	<0.005		0.08
	10	3	<0.005		0.38
	1	4	<0.005		0.12
	10	4	<0.005		0.64

*Analysis by AA.

†See leachate preparation in summary.

‡Not reported.

TABLE 26. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS* FBC LEACHATE SAMPLES (COLUMN)† (ppm)

Sample	Run	Hg	Sb	As
Bed reject material	3	<0.005	‡	0.79
	4	<0.005		3.2
FBC Cyclone #1	4	<0.005		1.6
FBC Cyclone #2	2	<0.005		1.5
	3	<0.005		<0.02
	4	<0.005		0.22

*Analysis by AA.

†See leachate preparation in summary.

‡Not reported.

TABLE 27. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS* METHOD 5 SAMPLES (ppm)

Sample	Run	Hg	Sb	As
Cyclone #1 (>4.6μ)	2	5.1	23	155
	3	0.76	9	159
	4	0.83	6	219
Cyclone #2 and filter (<4.6μ)	2	2.8	7.4	274
	3	1.0	7.3	253
	4	1.6	7.4	389
Impinger #1 and rinse† (H_2O_2)	2	0.069	<0.24	0.62
	3	0.39	<0.23	0.23
	4	0.24	<0.21	<0.09
Impinger #3 and #4 and† rinse (1 N HNO_3)	2	0.063	<0.24	<0.10
	3	0.12	<0.16	<0.10
	4	0.04	<0.06	<0.03

*Hg by flameless AAS, Sb by HGA/AAS, As by hydride generation AAS.

†Data from impinger samples reported in μg/m^3. HNO_3 used due to precipitate formation when using $(NH_4)_2\ S_2O_8/AgNO_3$.

TABLE 28. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS* FBC SAMPLES (ppm)

Sample	Run	Hg	Sb	As
Coal culm	2	0.21	0.7	34
	3	0.21	0.7	26
	4	0.29	1.2	40
Bed reject material	2	0.18	<0.5	35
	3	0.17	<0.5	41
	4	2.7	<0.5	36
FBC Cyclone #1	3	0.32	<0.5	35
	4	0.19	1.2	47
		----	----	----
FBC Cyclone #2	2	0.30	4.0	60
	3	0.36	4.8	90
	4	0.31	6.0	107
Blank		0.41	16.9	59

*Hg by flameless AAS, SB by HGA/AAS, As by hydride generation AAS.

TABLE 29. GAS CHROMATOGRAPHY* FOR INORGANIC GASES

Sample	SO_2 (ppm)	CO%	O_2%	CO_2%	N_2	H_2S (ppm)	COS (ppm)	NH_3 (ppm)	HCN (ppm)	CN_2
Run 1	379	.023	9.84	10.4	†	<2	<2	.13	<.06	†
Run 2	444	.04	9.40	11.3		<2	<2	.13	<.04	
Run 3	352	.01	11.5	9.6		<2.3	<2	.13	<.05	
Run 4	440	.016	10.6	10.4		<2.2	<2	.13	<.18	

*Gas chromatography was not used.

Analysis and collection techniques:

SO_2 by flame photometric--continuous withdrawal between cyclone #1 and #2.

CO/CO_2 by NDIR--continuous withdrawal from stream 21.

O_2 by paramagnetic--continuous withdrawal from stream 21.

H_2S by HRGM spectroscopy--collection by evacuated flask--stream 1.

COS by HRGM spectroscopy--collection by evacuated flask--stream 1.

NH_3 by Kjeldahl--collection by method 6 train with H_2SO_4 impinger--stream 1.

HCN by colorimetric--collection by method 6 train with KOH impinger--stream 1.

†Not reported.

TABLE 30. GAS CHROMATOGRAPHY* FOR INORGANIC GASES

Sample	SO_2 (ppm)	O_2%	CO_2%
Run 1	269	9.5 (12.75)	10.3 (7.25)
Run 2	609	9.2 (11.25)	11.0 (8.5)
Run 3	421	9.17 (11.25)	10.87 (8.8)
Run 4	511	8.6 (10.25)	10.8 (9.75)

*Gas chromatography was not used.

Analyses and collection techniques:

SO_2 by IC--collection by method 6 train--stream 1. Göksyr-Ross/IPA, Blank, H_2O_2, H_2O_2.

O_2CO_2 by Orsat--collection by Tedlar bag at stream 1 or 34. Data in parentheses refer to sample taken from stream 34.

TABLE 31. CHEMILUMINESCENCE FOR NO_x*

Sample	Units	NO_x concentrations
Run 1	ppm	228 (222)†
Run 2	ppm	193 (183)†
Run 3	ppm	248 (238)†
Run 4	ppm	239 (240)†

*Collection by continuous withdrawal at stream 21.
†NO concentrations.

TABLE 32. ANION ANALYSES OF FBC SAMPLES
(wt. percent)

Sample	Run	Cl	F	SO_4	SO_3	NO_3	NO_2	S	CO_3
Cyclone #1	2	0.001	0.22	0.45	0.20	<0.001	<0.0005	<0.01	0.06
	3	<0.001	0.22	1.47	----	<0.001	<0.0005	----	<0.03
	4	<0.001	0.20	1.47	----	<0.001	<0.0005	----	<0.03
Cyclone #2 and filters	2	<0.001	0.22	0.52	0.30	<0.001	<0.0005	0.01	0.01
	3	<0.001	0.29	1.24	0.20	<0.001	<0.0005	<0.01	<0.01
	4	<0.001	0.31	1.32	0.35	<0.001	<0.0005	<0.01	<0.01
Coal feed	2	0.029	0.037	----	----	----	----	----	0.05
	3	0.015	0.048	----	----	----	----	----	0.05
	4	0.029	0.033	----	----	----	----	----	0.10
Bed reject material	2	0.160	0.031	0.09	<0.01	0.003	<0.001	<0.01	0.10
	3	0.030	0.027	1.02	0.02	<0.001	<0.001	<0.01	0.10
	4	0.009	0.005	0.12	0.03	0.002	<0.001	<0.01	0.10
FBC cyclone #1	3	0.024	0.026	0.18	0.36	0.001	<0.001	<0.01	0.10
	4	0.013	0.045	0.09	0.26	<0.001	<0.001	<0.01	0.05
FBC cyclone #2	2	0.009	0.042	0.09	0.36	0.001	<0.001	<0.01	0.05
	3	0.060	0.065	0.57	0.13	<0.001	<0.001	<0.01	0.05
	4	0.011	0.081	0.39	0.15	<0.001	<0.001	<0.01	0.05
NBS Coal		0.030	0.010	1.02	<0.01	<0.001	<0.001	<0.01	0.35

Analysis methods:

Cl	Colormetric, ASTM D-512, method C.
F	Distillation/SPADNS, colorimetric, ASTM D1179, method A.
SO_4	Barium perchlorate/thorium titration, ASTM D516, method C.
SO_3	SO_2 evolution/West-Gaeke colorimetric, ASTM D2914.
NO_3	Brucine colorimetric, ASTM D992.
NO_2	Colorimetric, ASTM D1254
S	H_2S evolution/KIO_3 titration.
CO_3	CO_2 evolution, ASTM D1756.

TABLE 33. ANION ANALYSES OF FBC LEACHATES
ULTRASONIC SHAKING*
(ppm)

Sample	Run	Cl	F	SO_4	SO_3	NO_3	NO_2
Bed reject material	3-1	0.04	0.21	116	<0.5	<0.1	0.16
	3-10	18	5.0	137	<0.5	<0.1	0.16
	4-1	2.0	0.16	36	<0.5	<0.1	0.82
	4-10	3.0	0.64	8.3	<0.5	0.13	0.10
FBC cyclone #1	4-1	0.31	<0.05	93	<0.5	<0.1	<0.08
	4-10	0.23	0.09	<0.1	<0.5	<0.1	0.14
FBC cyclone #2	2-1	0.10	0.25	86	<0.5	<0.1	<0.05
	2-10	0.73	0.50	8.3	<0.5	<0.1	0.31
	3-1	1.4	1.3	1040	<0.5	0.35	<0.05
	3-10	0.72	1.5	<0.1	<0.5	<0.1	0.06
	4-1	1.5	10	773	<0.5	<0.1	<0.05
	4-10	0.04	0.90	<0.1	<0.5	<0.1	0.08

*See leachate preparation in summary. Analysis methods same as used in nonleached samples, table 32.

TABLE 34. ANION ANALYSES OF FBC LEACHATES
COLUMN*
(ppm)

Sample	Run	Cl	F	SO_4	SO_3	NO_3	NO_2
Bed reject material	3	2.3	1.1	334	<0.5	<0.1	1.0
	4	2.2	<0.05	198	<0.5	0.50	0.89
FBC cyclone #1	4	6.1	10	2260	<0.5	<0.1	<0.05
FBC cyclone #2	2	6.1	14	3440	<0.5	<0.1	0.05
	3	7.6	6.5	1590	<0.5	<0.1	0.05
	4	7.3	27	3010	<0.5	5.5	6.7

*See leachate preparation in summary.

Analysis methods same as used in nonleached samples, table 32.

TABLE 35. SSMS vs. OES COMPARISON*
(SSMS in ppmw, OES in weight percent)

		Method 5 Train								FBC					
		Cyclone 1		Cyclone 2		Coal Culm		Bed Ash		Cyclone 1		Cyclone 2		NBS	
Element	Run	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES
Si	2	~30%	18	~30%	18	~15%	15	~15%	15	---	---	~15%	25	~15%	25
	3	~30%	18	~30%	18	~15%	15	~15%	25	~15%	25	~15%	25	---	---
	4	~30%	18	~30%	18	~15%	15	~15%	25	~15%	25	~15%	25	---	---
Fe	2	~3%	5	~5%	9	~1%	2	5,000	2	~1%	---	1%	2	~1%	5
	3	~5%	5	~10%	9	5,000	2	5,000	2	5,000	2	1%	4	---	---
	4	~5%	5	~10%	9	~1%	2	5,000	2	1%	2	1%	4	---	---
Al	2	~10%	9	~10%	9	~15%	5	~15%	12	---	---	~15%	12	~15%	12
	3	~10%	9	~10%	9	~15%	5	~15%	12	~15%	12	~15%	12	---	---
	4	~10%	9	~10%	9	~15%	5	~15%	12	~15%	12	~15%	12	---	---
K	2	~2%	4	5,000	4	~3%	2.5	~10%	5	---	---	~3%	4	~2%	---
	3	5,000	4	~1%	4	~2%	2.5	~10%	4	~5%	4	~5%	4	---	---
	4	~1%	4	5,000	4	~1%	2.5	~10%	5	~5%	4	~5%	4	---	---
Ca	2	3,000	0.1	2,000	0.1	300	0.02	5,000	0.05	---	---	3,000	0.05	~5%	5-7
	3	1,000	0.1	3,000	0.1	300	0.02	~1%	1	5,000	0.1	3,000	0.2	---	---
	4	2,000	0.05	2,000	0.05	200	0.02	~1%	0.5	5,000	0.03	5,000	0.05	---	---
Mg	2	~1%	0.3	~1%	0.3	2,000	0.2	~1%	0.3	---	---	~1%	0.3	~1%	1
	3	5,000	0.3	~1%	0.3	2,000	0.2	5,000	0.3	2,000	0.3	~1%	0.3	---	---
	4	5,000	0.3	~1%	0.3	1,000	0.2	~1%	0.3	~1%	0.3	~1%	0.3	---	---
Ti	2	~2%	1	~2%	1	3,000	0.3	3,000	0.5	---	---	3,000	0.5	3,000	0.3
	3	~2%	1	~2%	1	2,000	0.3	3,000	0.5	3,000	0.5	3,000	0.5	---	---
	4	~1%	1	~3%	1	3,000	0.3	5,000	0.5	3,000	0.5	3,000	0.5	---	---
Na	2	3,000	0.1	3,000	0.1	2,000	0.1	3,000	0.1	---	---	2,000	0.1	2,000	0.1
	3	1,000	0.1	1,000	0.1	500	0.1	2,000	0.1	1,000	0.1	2,000	0.1	---	---
	4	2,000	0.1	3,000	0.1	300	0.1	2,000	0.1	1,000	0.1	3,000	0.1	---	---
Pb	2	300	0.01	100	0.02	3	<.01	100	<.01	---	---	100	<.01	30	0.01
	3	200	0.02	100	0.02	3	<.01	100	<.01	30	<.01	200	0.02	---	---
	4	500	0.02	100	0.02	3	<.01	100	<.01	30	<.01	30	0.02	---	---

*OES valid to ± 50%.

TABLE 35 (con.)

Element	Run	Method 5 Train				Coal Culm		Bed Ash		FBC				NBS	
		Cyclone 1		Cyclone 2						Cyclone 1		Cyclone 2			
		SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES
Ba	2	2,000	0.04	1,000	0.06	2,000	0.02	3,000	0.04	---	---	2,000	0.03	3,000	0.01
	3	500	0.04	2,000	0.06	1,000	0.02	500	0.03	2,000	0.03	2,000	0.03	---	---
	4	500	0.04	2,000	0.04	2,000	0.02	1,000	0.04	1,000	0.03	500	0.03	---	---
Mn	2	100	0.007	200	0.01	30	0.003	30	0.005	---	---	50	0.003	200	0.03
	3	50	0.007	200	0.01	20	0.003	30	0.01	30	0.003	30	0.003	---	---
	4	100	0.007	200	0.01	10	0.003	20	0.005	30	0.003	20	0.003	---	---
V	2	300	0.02	300	0.04	200	0.005	100	0.01	---	---	200	0.01	100	0.01
	3	500	0.03	1,000	0.04	100	0.005	100	0.01	100	0.01	200	0.01	---	---
	4	300	0.03	500	0.03	100	0.005	100	0.01	50	0.01	50	0.01	---	---
Cu	2	300	0.007	100	0.007	10	<.003*	50	<.003*	---	---	30	<.003*	100	0.01
	3	300	0.007	100	0.007	5	<.003*	10	<.003*	50	<.003*	30	0.003	---	---
	4	100	0.007	100	0.007	5	<.003*	30	<.003*	20	<.003*	30	0.003	---	---
Zr	2	500	0.03	500	0.03	200	0.01	100	0.02	---	---	200	0.02	100	0.02
	3	500	0.03	500	0.03	50	0.01	50	0.02	200	0.02	100	0.02	---	---
	4	500	0.03	500	0.03	200	0.01	100	0.02	100	0.02	100	0.02	---	---
Ni	2	200	0.005	200	0.007	50	<.01	20	<.01	---	---	50	<.01*	100	.01
	3	100	0.005	200	0.007	50	<.01	20	<.01	30	<.01*	50	<.01*	---	---
	4	100	0.005	200	0.007	100	<.01	10	<.01	30	<.01*	50	<.01*	---	---
Cr	2	500	0.01	300	0.02	50	≤.01*	50	≤.01*	---	---	30	≤.01*	10	≤0.01*
	3	300	0.01	300	0.02	30	≤.01*	10	≤.01*	50	≤.01*	30	0.01	---	---
	4	500	0.01	300	0.02	100	≤.01*	20	≤.01*	30	≤.01*	20	0.01	---	---
Sr	2	300	0.01	200	0.01	100	<.01	300	<.01	---	---	100	<.01	2,000	0.1
	3	200	0.01	300	0.01	100	<.01	100	<.01	100	<.01	100	<.01	---	---
	4	200	0.01	200	0.01	200	<.01	100	<.01	100	<.01	300	<.01	---	---
B	2	5	---	20	---	20	<.01	20	<.01	---	---	30	<.01	50	0.03
	3	5	---	20	---	50	<.01	50	<.01	10	<.01	30	<.01	---	---
	4	10	---	20	---	20	<.01	100	<.01	20	<.01	30	<.01	---	---

*Trace.

TABLE 35 (con.)

		Method 5 Train								FBC					
		Cyclone 1		Cyclone 2		Coal Culm		Bed Ash		Cyclone 1		Cyclone 2		NBS	
Element	Run	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES	SSMS	OES
Co	2	30	---	50	---	2	<.01	5	<.01	---	---	10	<.01	30	<0.01*
	3	30	---	50	---	2	<.01	5	<.01	10	<.01	10	<.01	---	---
	4	30	---	50	---	10	<.01	3	<.01	5	<.01	10	<.01	---	---

*Trace.

TABLE 36. LC FRACTIONATION
COAL CULM FROM FEED HOPPER

Fraction	Run 2				Run 3			
	TCO	Grav μg/g	Total	Total	TCO	Grav μg/g	Total	Total
LC 1		112.6				83.6		
LC 2		0				0		
LC 3		5.2				3.0		
LC 4		5.2				4.0		
LC 5		0				0.6		
LC 6		0				10.0		
LC 7		13.6				4.2		
LC 8		0.8				2.4		

Fraction	Run 4			
	TCO	Grav μg/g	Total	Total
LC 1		131.8		
LC 2		0		
LC 3		6.8		
LC 4		3.0		
LC 5		0		
LC 6		13.0		
LC 7		56.0		
LC 8		LOST		

NOTE: Values corrected for solvent blank A.

TABLE 37. LC FRACTIONATION
BED REJECT MATERIAL

Fraction	Run 2				Run 3			
	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		11.4				13.2		
LC 2		0				0		
LC 3		0.6				1.8		
LC 4		4.8				3.8		
LC 5		0				0		
LC 6		4.4				1.4		
LC 7		21.8				44.4		
LC 8		2.8				0		

Fraction	Run 4			
	TCO	Grav µg/g	Total	Total
LC 1		3.4		
LC 2		0		
LC 3		0		
LC 4		2.6		
LC 5		0		
LC 6		4.4		
LC 7		0		
LC 8		2.6		

NOTE: Values correlated for solvent blank A.

TABLE 38. LC FRACTIONATION
FBC CYCLONE NUMBER 1

	Run 3				Run 4			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		14.2				3.4		
LC 2		0				0		
LC 3		0				1.2		
LC 4		4.0				1.6		
LC 5		0.8				0		
LC 6		7.8				0.6		
LC 7		0.8				0		
LC 8		0				10.8*		

*Some silica gel in sample.

Note: Values corrected for solvent blank A.

TABLE 39. LC FRACTIONATION
FBC CYCLONE NUMBER 2

	Run 2				Run 3			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		8.2				0		
LC 2		0				0		
LC 3		1.2				3.8		
LC 4		2.0				0.6		
LC 5		0				0		
LC 6		0.4				3.0		
LC 7		5.2				5.4		
LC 8		0				0.6		

	Run 4			
Fraction	TCO	Grav µg/g	Total	Total
LC 1		2.2		
LC 2		0		
LC 3		3.0		
LC 4		2.6		
LC 5		0		
LC 6		3.0		
LC 7		12.2		
LC 8		2.6		

NOTE: Values corrected for solvent blank A.

TABLE 40. LC FRACTIONATION
HVSS FILTER

	Run 2				Run 3			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		52.0				50.0		
LC 2		4.0				0		
LC 3		0				0		
LC 4		0				0		
LC 5		0				0		
LC 6		0				0		
LC 7		0				0		
LC 8		0				0		

	Run 4			
Fraction	TCO	Grav µg/g	Total	Total
LC 1		11.0		
LC 2		0		
LC 3		0		
LC 4		0		
LC 5		0		
LC 6		32.0		
LC 7		0		
LC 8		0		

Values corrected for solvent blank C.

TABLE 41. LC FRACTIONATION
TENAX EXTRACT

	Run 2				Run 3			
Fraction	TCO	Grav	Total	Total µg/m³	TCO	Grav	Total	Total µg/m³
LC 1				410*				67.9*
LC 2				97.3				203.7
LC 3				3.8				4670.2†
LC 4				610.1†				37.4
LC 5				70.2				5.8
LC 6				22.6				79.9
LC 7				493.4				46.8
LC 8				12.9				0.3

	Run 4			
Fraction	TCO	Grav	Total	Total µg/m³
LC 1				30.3*
LC 2				29.7
LC 3				1159.7†
LC 4				101.5
LC 5				27.6
LC 6				17.3
LC 7				122.7
LC 8				32.4

*Silicone grease detected in sample.

†High weights possibly result of Tenax breakdown with quinone formation.

NOTE: Values corrected for Tenax blank.

TABLE 42. LC FRACTIONATION CH_2Cl_2 RINSE OF METHOD 5 TRAIN

	Run 2				Run 3			
Fraction	TCO	Grav	Total	Total µg/m^3	TCO	Grav	Total	Total µg/m^3
LC 1				110.8				45.8
LC 2				62.5				79.6
LC 3				0				0
LC 4				39.3				0
LC 5				0				0
LC 6				0				0
LC 7				143.8				127.4
LC 8				12.8				0

	Run 4			
Fraction	TCO	Grav	Total	Total µg/m^3
LC 1				90.6
LC 2				24.8
LC 3				0
LC 4				0
LC 5				0
LC 6				0.9
LC 7				83.6
LC 8				30.3

Values corrected for solvent C.

TABLE 43. LC FRACTIONATION

	Solvent Blank A				Tenax Blank			
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		3.0						113.0
LC 2		12.2						79.0
LC 3		2.6						191.0
LC 4		1.6						378.0
LC 5		8.2						61.0
LC 6		2.2						34.0
LC 7		7.6						208.0
LC 8		2.0						62.0

	Solvent Blank C			
Fraction	TCO	Grav µg/g	Total	Total
LC 1		49.0		
LC 2		32.0		
LC 3		547.0		
LC 4		93.0		
LC 5		112.0		
LC 6		117.0		
LC 7		170.0		
LC 8		34.0		

TABLE 44. IR REPORT
SAMPLE: COAL CULM

LC	Run 2	Run 3	Run 4
1	---	---	---
2	---	---	---
3	Ester	---	Ester
4	Ester	Ester, possibly aromatic; may be methyl	---
5	---	Phthalate ester	Possibly alcohol or mixture of alcohol
6	---	Ketone, may be aromatic; phthalate ester	---
7	---	---	Inorganic substances
8	---	---	Lost in liquid chromatography

Note: Wavelength, intensity and comments were not detailed in this study. A dash (---) indicates no compounds were seen that were not present in the blank.

TABLE 45. IR REPORT
SAMPLE: BED REJECT MATERIAL

LC	Run 2	Run 3	Run 4
1	Saturated hydrocarbons--long chain	Saturated hydrocarbons--long chain	Saturated hydrocarbons--long chain
2	---	---	---
3	---	Ester	---
4	Ester	---	Ester
5	---	---	Phthalate ester
6	---	---	Phthalate ester
7	---	Aliphatic ester and possible carboxylic acid	Aliphatic ester and possible carboxylic acid
8	Possible sulfonic acid	---	---

TABLE 46. IR REPORT
SAMPLE: FBC CYCLONE NUMBER 1 ASH

LC	Run 3	Run 4
1	Saturated hydrocarbons	Saturated hydrocarbons--long chain
2	---	---
3	Phthalate ester	Trace ester
4	---	Phthalate ester
5	---	---
6	---	---
7	---	---
8	---	---

TABLE 47. IR REPORT
SAMPLE: FBC CYCLONE NUMBER 2 ASH

LC	Run 2	Run 3	Run 4
1	Saturated unbranched hydrocarbon	---	Saturated unbranched hydrocarbon
2	---	---	---
3	---	Trace ester	Phthalate ester
4	Ester, not an acetate	---	Ester, probably unsaturated
5	Ester, not an acetate	---	---
6	Ester not an acetate	Possibly organic phosphate or ester	---
7	---	---	---
8	---	---	---

TABLE 48. IR REPORT
SAMPLE: METHOD 5 TENAX COLUMN

LC	Run 2	Run 3	Run 4	Blank
1	Silicone grease Aromatic hydrocarbon	Silicone grease Aliphatic hydrocarbon	Silicone grease Aliphatic hydrocarbon	Normal hydrocarbon background
2	Silicone grease Aromatic hydrocarbon, ester	Silicone grease Aromatic ester	Silicone grease Ester	Aromatic ester
3	Silicone grease Aromatic ester Quinone[*]	Quinone?	Quinone, ketone? Phenol?	Quinone (like run 3, LC 3), aromatic ester (like run 3, LC 2)
4	Quinone[*]	Quinone?	Quinone, ketone? Phenol?	Quinone (like run 3, LC 3), ketone II
5	Aromatic ketone acrylate	Inorganic[‡]	Weak spectrum (like fraction 4)	Quinone (like run 3, LC 3), ketone II
6	Aromatic ketone acrylate	Inorganic[‡]	Below IR limits	Clean
7	Cyclic ketone[†]	Ketone I, alcohol?, aromatic[§], ether	Aromatic phenol? Aromatic aldehyde?	Similar to run 3, LC 7 with alcohol
8	Polar material Aliphatic ester	Ketone I, alcohol?, aromatic[§], ether	Polar materials	Same as fraction 7

[*] Quinone is probably a contaminant either from sampling column or sample handling.
[†] Cyclic ketone may be a substituted cyclohexanone.
[‡] Possible fused hetrocyclic aromatic with nitrogen or sulfur.
[§] Aromatic may be associated with the ketone, alcohol, or the possible ether.

TABLE 49. IR REPORT
SAMPLE: METHOD 5 PARTICULATES FROM STREAM NUMBER 1

LC	Run 2	Run 3	Run 4
1	Blank	Aliphatic hydrocarbon, aromatic hydrocarbon?	Aliphatic hydrocarbon, unsaturated hydrocarbon
2	Mix of possible polynuclear aromatic ester and aliphatic ester	Ester mix similar to run 2, fraction 2	Aromatic hydrocarbon, aliphatic hydrocarbon, ester
3	Mix of possible polynuclear aromatic ester and aliphatic ester	Aliphatic ester of run 2, fraction 2, and ketone	Mix of ester as in run 2, fraction 2
4	Same aliphatic ester as in fractions 2 and 3	Aliphatic ester of run 2, fraction 2, and ketone	Aliphatic ester of run 2, fraction 2
5	Same aliphatic ester as in fractions 2 and 3	Aliphatic ester of run 2, fraction 2, and ketone	Aliphatic ester of run 2, fraction 2
6	Same aliphatic ester as in fractions 2 and 3	Similar to fractions 3, 4, and 5, but with more ketone	Blank
7	Ketone, possibly quinone	Carboxylic acid, possible ketone	Ketone, carboxylic acid, same ester as run 3, fraction 7
8	Ester, possibly that in fractions 4, 5, and 6	Blank	Small amount of polar material

TABLE 50. IR REPORT
SAMPLE: HVSS FILTER FROM STREAM 34

LC	Run 2	Run 3	Run 4
1	Aliphatic hydrocarbon	Aliphatic hydrocarbon, aromatic hydrocarbon	Aliphatic hydrocarbon
2	Ester I	Ester II	Blank*
3	Ester I	Ester II	Blank*
4	Blank*	Blank*	Blank*
5	Blank*	Blank*	Blank*
6	Blank*	Blank*--maybe trace of ketone	Blank*
7	Ketone†	Ketone†	Ketone†
8	Blank*	Blank*	Blank*

*Blank implied below IR detection limits with only normal background spectra present.
†Ketone in fraction 7 of all three samples are possibly the same, but not definitely.

STUDY NUMBER 12

DATA SOURCE:

METHOD FOR ANALYZING EMISSIONS FROM ATMOSPHERIC FLUIDIZED-BED COMBUSTOR

EPA-600/7-77-034

DATA STATUS: Final Report, April 1977

AUTHORS: E. L. Merryman, A. Levy, G. W. Felton, K. T. Liu, J. M. Allen, and H. Nack

CONTRACTOR:
Battelle-Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201
Contract No. 68-02-1409, Task 33
Program Element No. EHB536

TASK OFFICER:
Walter B. Steen
Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

GENERAL

This manual gives data resulting from sampling and analysis of FBC combustion units. The 6-in. FBC unit at BCL was studied as an example, and six high priority sampling sites on the unit (of a possible 30 effluent streams) were selected for this study: (1) coal feed stream, (2) limestone feed stream, (3) overflow bed material, (4) ash, (5) sludge (from water scrubber to flue gas stack), and (6) flue gas stream (sampled at several different sites, 6-10). Figure 1 (from the text), shows the sampling locations schematically. Samples from the three runs were taken after stabilized FBC conditions had been achieved; Table 1, from the text, enumerates the samples taken.

Firing conditions for the three runs were as follows:

	Run 1	Run 2	Run 3
Coal feed rate, lb/h	15.9	8.8	9.3
Limestone feed rate, lb/h	15.4	4.3	8.1
Air feed rate, lb/h	145.0	87.3	84.2
Bed height: expanded, in.	48	48	48
settled, in.	12.6	21.6	21.6
Bed temperature, °F	1,538	1,655	1,490
Superficial gas velocity, ft/s	9.1	6.0	5.3
Ca/S ratio	6.7:1	2.9:1	7.1:1
Particulate loading, g/m^3	1.44	1.64	NA

Runs 1 and 2 were selected for analysis since analytical techniques were somewhat different between the two runs (see attached Table 2 from the text). Analytical results were presented comparing this site with other FBC units and with pulverized coal units. The FBC was higher in CO (2,090 v. 90-300 ppm), but lower in SO_2 (730 v. 1,200-1,500 ppm) than a pulverized coal operation.

It was suggested for FBC units that additional sampling programs be included for biological testing, radioactivity measurements, and noise measurements.

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GASEOUS GRAB

Continuous monitors already installed at the FBC site were used for measuring CO, CO_2, SO_2, NO_x, O_2, and total hydrocarbons. Pyrex glass wool plugs used to remove particulates and water vapors from gaseous streams may have caused some bias in data. Method 5-type sorbtion trains were used to sample for acidic (HCl, HF, HCN) and basic (NH_3) gaseous components. A Göksoyr-Ross apparatus was used to sample for SO_3.

SASS

No SASS train was used. A modified Method 5 train was used to sample particulate-containing gaseous streams. Modifications included addition of a heated small glass cyclone (which collects particles >2.3 μm) prior to the filter (which collects particles 2.3 μm - 0.1 μm) and addition of a Tenax-GC cartridge downstream of the filter. Sampling was isokinetic but from a single, representative point (no traverse). The sampling point was downstream of a process cyclone that removed particles >27 μm. A Method 5-type sorbtion train containing an oxidizing solution was used to collect certain trace metals (Pb, Hg, Se, Te, Be, As, and Cd). POM analysis was run on the FBC cyclone particulates and flue gas vapors adsorbed on Tenax.

FUGITIVE EMISSIONS

Sampling was not performed in this study.

LIQUIDS AND SLURRIES

The solid material (sludge) caught in the water scrubber from the FBC flue gas stack was separated from the scrubber liquids by centrifugation. The solids were dried and stored in the dark. Solids were subsequently analyzed for trace metals, organic classes, $SO_4^{=}$, $SO_3^{=}$, and via proximate/ultimate analysis for C, H, N, S, O, ash, volatiles, and moisture.

SOLIDS

Three to four cross-sectional grab samples were collected over a 2-hour period from the coal-feed conveyor belt, and these were combined in a single plastic container, sealed, and stored in the dark. This composite sample was analyzed for trace metals by SSMS and for Na and Ca by AA. In addition to Level I requirements, proximate/ultimate analysis (ASTM-D-291), total sulfur forms (ASTM-D2492-68), particle size (sieve), and heating value (ASTM-D2015-66) were run on the composite coal sample.

Limestone sorbent was sampled and composited like the coal. The limestone was tested for trace metals (SSMS), selected anions (Ca, Mg, CO_3, NO_2, NO_3, by spectrophotometric methods), and particle size (by sieve).

A representative sample of overflow bed material, which was primarily limestone, was taken from the overflow container at the end of a 2-hour run. This material was analyzed for trace metals by SSMS, anions ($SO_4^{=}$, $SO_3^{=}$, $S^{=}$, NO_2^{-}, NO_3^{-}) by wet chemical methods, organics by extraction--LC-IR, and particle size by sieve. Particles less than 325 mesh were classified as bed ash, and the same analyses were run on these as on the overflow bed materials.

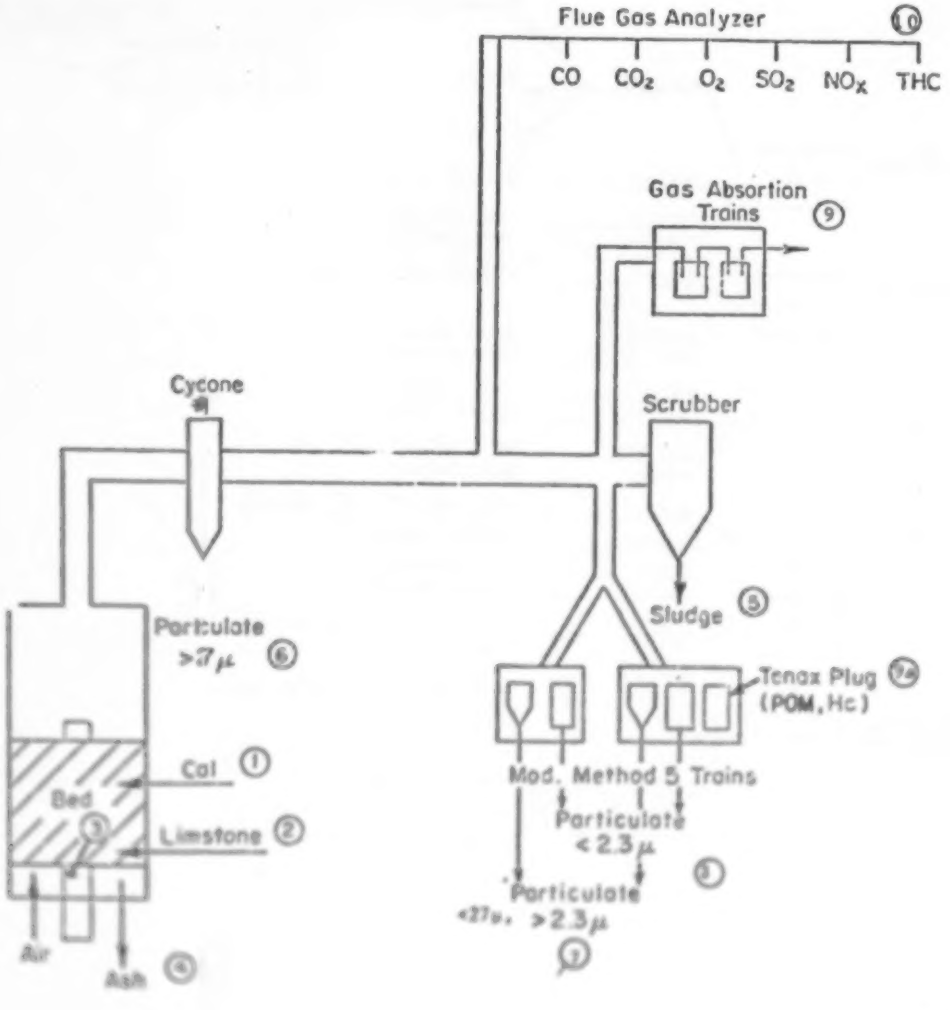
Flue Gas Analyzer ⑩
CO
CO₂
O₂
SO₂
NOₓ
THC
Gas Absorption Trains ⑨
Cycone
Scrubber
Sludge ⑤
Particulate >7μ ⑥
Tenax Plug (POM, Hc) ⑦a
Cal ①
Bed
③
Limstone ②
Mod. Method 5 Trains
Particulate <2.3μ ⑧
Particulate <7μ, >2.3μ ⑦
Air
Ash ④

Figure 1. Schematic outline of fluidized-bed combustor and sampling locations.

TABLE 1. SAMPLE IDENTIFICATION AND ANALYSES

Material	Fluid-Bed Sample No.	Sample Number Designation		Analyses
Illinois #6 coal (-8 M)	S-2-1	S-2-1-1	Proximate/ultimate	(1) Moisture (2) Ash (3) C,H,N,S, O(by difference) (4) Volatile matter (5) Fixed carbon (by difference)
			Sulfur	(1) Total (2) Pyrites (3) Organic (4) SO_4
			Na, Ca	
		S-2-1-2	Heating value	
		S-2-1-3	Trace metals	
		S-2-1-4	Particle Size	
Grove limestone	S-2-2	S-2-2-1	Ca, Mg, $CO_3^=$, NO_2^-, NO_3^-	
		S-2-2-2	Trace metals	
		S-2-2-3	Particle Size	
Bed material	S-2-3	S-2-3-1	Trace metals	
		S-2-3-4	Organic Classes	
		S-2-3-2	$SO_4^=$, $SO_3^=$, $S^=$, NO_2^-, NO_3^-	
		S-2-3-3	Particle Size	
Ash (-325 M)	S-2-4	S-2-4-1	NO_2^-, NO_3^-, $S^=$, $SO_4^=$, and $SO_3^=$	
		S-2-4-2	Fusion temperature	
		S-2-4-3	Trace metals	
		S-2-4-4	Organic Classes	
Sludge	S-2-5	S-2-5-1	C, H, N, S, O, $SO_4^=$, and $SO_3^=$	
		S-2-5-2	Trace Metals	
		S-2-5-3	Organic Classes	
Flue gas stream Particulate >27μ	S-2-6	S-2-6-1	Trace metals (approx 60 metals)	
		S-2-6-2	C, H, N, S, O	
		S-2-6-3	Anions, NO_2^-, NO_3^-, $SO_4^=$, $SO_3^=$	
		S-2-6-4	Organic classes	
			POM	
			Organic and reduced sulfur compounds	
		S-2-6-5	Partical Size	

TABLE 1 (con.)

Material	Fluid-Bed Sample No.	Sample Number Designation	Analyses
Flue gas stream Particulate <27μ (If two particulate fractions involved here, e. g. <27 >2.3 , and <2.3 , smaller fraction is designated Batch No. S-2-8 and same analyses as S-2-7.)	S-2-7	S-2-7-1	Trace metals
		S-2-7-2	C, H, N, S, O
		S-2-7-3	Anions, $CO_3^=$, NO_2^-, NO_3^-, $SO_4^=$, $SO_3^=$
		S-2-7-4	Organic classes POM Organic and reduced sulfur compounds
		S-2-2-5	Particle Size
Flue gas stream Gases	S-2-9	(AL-3) S-2-9-1	Organic classes POM Organics - reduced sulfur
		S-2-9-2 (AL-2)	HCl, HF
		S-2-9-3 (AL-2)	HCN
		S-2-9-4 (AL-2)	NH_3
		S-2-9-5 (AL-4)	Trace elements (solution)
		S-2-9-6 (AL-3)	SO_3 (Goksoyr-Ross)
Gases, continuous monitoring	S-2-10	S-2-10-1	O_2
		S-2-10-2	CO_2
		S-2-10-3	CO
		S-2-10-4	SO_2
		S-2-10-5	NO_x
		S-2-10-6	HC

TABLE 2. CHANGES MADE BETWEEN RUN NOS. 1 and 2

Change Mode	Reason for Change
1. Proximate/ultimate analysis made on bed material -325 M in Run No. 1; discontinued in Run No. 2	Analysis of little use
2. Fe, Al, Si, K, Cl^-, F^- analyzed individually (e.g., atomic absorption, ion electrode, etc.) in bed material samples in Run No. 1; determined these elements by SSMS in Run No. 2	More efficient, eliminate duplication
3. NO_3^-, $SO_3^=$, $S^=$, and $CO_3^=$ anions added to the analysis of bed material and particulate samples in Run No. 2	More completely define anion concentrations in solid samples
4. Analysis for POM increased to include bed material, sludge, and particulate samples in Run No. 2	More completely define the POM levels in solid samples
5. Organic class analyses increased to include bed material and sludge samples in Run No. 2	More completely define organic classes in solid samples
6. Increased analysis of trace elements to include sludge samples in Run No. 2	To better define trace elements in effluent waste material
7. Increased particle size analysis to include bed material and particulate (both >27 and <27 microns) samples in Run No. 2	To define particle size ranges more completely
8. Reduced quantity of solid sample for bioassay from 20 grams in Run No. 1 to 2 grams in Run No. 2	Only 2 grams of sample needed for analysis

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY--RUN 1
ILLINOIS NUMBER 6 COAL
(ppmw)

U	1.0	Dy	0.70	Rh	<0.1	Cr	13
Th	1.7	Tb	*	Ru	<0.1	V	20
Bi	*	Gd	0.40	Mo	2.9	Ti	240
Pb	0.47	Eu	*	Nb	3.1	Sc	1.6
Tl	0.59	Sm	0.71	Zr	10	Ca	~4,000 (0.41)†
Hg	0.16	Nd	6.6	Y	9.4	K	~1,200
Au	<0.1	Pr	2.1	Sr	80	Cl	~1,100
Pt	<0.1	Ce	9.8	Rb	21	S	>0.5%
Ir	<0.1	La	5.0	Br	4.7	P	3.2
Os	<0.1	Ba	35	Se	0.29	Si	>1%
Re	‡	Cs	0.65	As	2.7	Al	>1%
W	0.31	I	0.10	Ge	2.0	Mg	400 (0.06)†
Ta	0.27	Te	<0.37	Ga	1.7	Na	890 (0.23)†
Hf	0.18	Sb	0.56	Zn	37	F	110
Lu	<0.1	Sn	0.28	Cu	9.0	B	93
Yb	0.23	In	‡	Ni	33	Be	0.17
Tm	<0.1	Cd	0.10	Co	4.0	Li	0.13
Er	0.34	Ag		Fe	>1%		
Ho	<0.1	Pd	<0.1	Mn	80		

*Not reported.
†Weight percent by AA.
‡Internal standard.

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY--RUN 1
GROVE LIMESTONE
(ppmw)

U	0.34	Dy	0.20	Rh	<0.1	Cr	0.87
Th	0.34	Tb	*	Ru	<0.1	V	7
Bi	<0.24	Gd	*	Mo	0.67	Ti	87
Pb	0.86	Eu	*	Nb	0.32	Sc	<0.11
Tl	*	Sm	0.31	Zr	7.0	Ca	>1% (37.1)†
Hg	<0.01	Nd	1.7	Y	1.7	K	>0.5%
Au	<0.1	Pr	0.86	Sr	470	Cl	18
Pt	<0.1	Ce	2.9	Rb	7.3	S	240
Ir	<0.1	La	2.2	Br	1.2	P	140
Os	<0.1	Ba	90	Se	*	Si	<1%
Re	‡	Cs	0.19	As	<0.61	Al	~3,000
W	<0.22	I	2.9	Ge	*	Mg	<0.5 (0.51)†
Ta	0.33	Te	*	Ga	0.23	Na	140
Hf	1.8	Sb	*	Zn	4.0	F	140
Lu	<0.15	Sn	0.12	Cu	2.2	B	6.2
Yb	<0.21	In	‡	Ni	0.69	Be	<0.18
Tm	<0.1	Cd	*	Co	0.14	Li	2.9
Er	*	Ag	*	Fe	~1,200		
Ho	<0.1	Pd	<0.1	Mn	15		

*Not reported.
†Weight percent by AA.
‡ Internal standard.

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY--RUN 1
BED MATERIAL
(ppmw)

U	0.80	Dy	0.49	Rh	<0.1	Cr	1.3
Th	1.1	Tb	*	Ru	<0.1	V	7
Bi	<0.24	Gd	*	Mo	1.2	Ti	130
Pb	1.9	Eu	0.25	Nb	0.69	Sc	0.16
Tl	*	Sm	0.83	Zr	28	Ca	>1%
Hg	<0.01	Nd	3.8	Y	3.6	K	~1,600
Au	<0.1	Pr	2.0	Sr	470	Cl	120
Pt	<0.1	Ce	15	Rb	7.3	S	>0.5%
Ir	<0.1	La	6.4	Br	4.5	P	310
Os	<0.1	Ba	180	Se	*	Si	>0.5%
Re	†	Cs	0.43	As	0.67	Al	~3,000
W	<0.32	I	0.29	Ge	1.4	Mg	<0.5%
Ta	0.87	Te	*	Ga	1.1	Na	320
Hf	1.1	Sb	*	Zn	17	F	170
Lu	<0.15	Sn	0.55	Cu	78	B	33
Yb	<0.49	In	†	Ni	23	Be	<0.18
Tm	<0.1	Cd	0.14	Co	0.14	Li	5.7
Er	0.11	Ag	*	Fe	~4,600		
Ho	<0.1	Pd	<0.1	Mn	26		

*Not reported.
†Internal standard.

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY--RUN 1
ASH >27 μm
(ppmw)

U	4.0	Dy	2.0	Rh	<0.1	Cr	32
Th	5.7	Tb	0.25	Ru	<0.1	V	38
Bi	<0.24	Gd	0.32	Mo	3.3	Ti	870
Pb	7.2	Eu	0.50	Nb	3.2	Sc	0.73
Tl	1.0	Sm	1.7	Zr	35	Ca	>1%
Hg	0.01	Nd	7.5	Y	8.3	K	<0.5%
Au	<0.1	Pr	2.0	Sr	140	Cl	240
Pt	<0.1	Ce	11	Rb	37	S	310
Ir	<0.1	La	6.4	Br	12	P	310
Os	<0.1	Ba	180	Se	4.4	Si	>1%
Re	†	Cs	4.4	As	2.0	Al	>1%
W	0.36	I	1.2	Ge	3.0	Mg	>0.5%
Ta	0.43	Te	*	Ga	2.3	Na	~3,200
Hf	0.79	Sb	0.67	Zn	80	F	290
Lu	0.38	Sn	0.92	Cu	78	B	710
Yb	1.6	In	†	Ni	46	Be	18
Tm	<0.1	Cd	0.67	Co	14	Li	220
Er	0.28	Ag	0.30	Fe	>1%		
Ho	0.39	Pd	<0.1	Mn	210		

†Internal standard.
*Not reported.

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY--RUN 1
PARTICULATES >27 μm
(ppmw)

U	1.7	Dy	1.4	Rh	<0.1	Cr	15
Th	2.4	Tb	0.19	Ru	<0.1	V	25
Bi	<0.24	Gd	0.32	Mo	3.3	Ti	870
Pb	4.3	Eu	0.50	Nb	3.2	Sc	1.6
Tl	0.48	Sm	1.7	Zr	35	Ca	>1%
Hg	<0.01	Nd	10	Y	3.6	K	>0.5% (0.47)†
Au	<0.1	Pr	2.0	Sr	470	Cl	240
Pt	<0.1	Ce	15	Rb	20	S	310
Ir	<0.1	La	6.4	Br	6.0	P	310
Os	<0.1	Ba	180	Se	0.94	Si	>1% (6.00)†
Re	‡	Cs	1.9	As	4.1	Al	>1% (2.53)†
W	<0.32	I	0.58	Ge	3.0	Mg	>0.5%
Ta	0.87	Te	*	Ga	2.3	Na	~1,400
Hf	1.8	Sb	0.67	Zn	67	F	290
Lu	0.50	Sn	1.2	Cu	66	B	500
Yb	1.6	In	‡	Ni	23	Be	2.6
Tm	<0.1	Cd	0.67	Co	2.1	Li	*
Er	0.28	Ag	*	Fe	>1% (5.42)*		
Ho	0.16	Pd	<0.1	Mn	60		

†Weight percent by AA.
‡Internal standard.
*Not reported.

TABLE 8. SPARK SOURCE MASS SPECTROSCOPY--RUN 1
PARTICULATES <27 μm
(ppmw)

U	8.0	Dy	2.0	Rh	<0.1	Cr	87
Th	11	Tb	0.48	Ru	<0.1	V	140
Bi	0.80	Gd	0.69	Mo	14	Ti	~2,200
Pb	43	Eu	0.94	Nb	16	Sc	7.3
Tl	10	Sm	3.6	Zr	120	Ca	>1%
Hg	14	Nd	21	Y	29	K	>1% (2.10)†
Au	<0.1	Pr	15	Sr	250	Cl	300
Pt	<0.1	Ce	44	Rb	37	S	310
Ir	<0.1	La	30	Br	60	P	310
Os	<0.1	Ba	320	Se	9.4	Si	>1% (11.0)†
Re	‡	Cs	4.3	As	6.1	Al	>1% (7.36)†
W	1.1	I	29	Ge	14	Mg	>0.5%
Ta	0.87	Te	*	Ga	11	Na	>0.5%
Hf	1.8	Sb	1.4	Zn	140	F	450
Lu	0.50	Sn	12	Cu	120	B	~2,000
Yb	2.5	In	‡	Ni	99	Be	6.0
Tm	0.14	Cd	1.0	Co	14	Li	57
Er	0.56	Ag	0.30	Fe	>1% (6.68)*		
Ho	0.61	Pd	<0.1	Mn	60		

*Not reported.
†Weight percent by AA.
‡Internal standard.

TABLE 9. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
ILLINOIS NUMBER 6 COAL
(ppmw)

U	<0.5	Dy	<1	Rh	<0.5	Cr	100
Th	<1	Tb	<0.2	Ru	<1	V	500
Bi	<0.5	Gd	<1	Mo	30	Ti	5,000
Pb	<1	Eu	<0.5	Nb	3	Sc	3
Tl	<1	Sm	<1	Zr	300	Ca	5,000
Hg	<2	Nd	<2	Y	30	K	~0.6%
Au	<0.5	Pr	5	Sr	500	Cl	~0.5%
Pt	<1	Ce	50	Rb	20	S	5,000
Ir	<1	La	20	Br	10	P	100
Os	<1	Ba	200	Se*	<5	Si	~20%
Re	<1	Cs	3	As	<3	Al	~5%
W	<1	I	3	Ge	100	Mg	~2%
Ta	<0.5	Te	<1	Ga*	<5	Na	~2%
Hf	<2	Sb	<0.5	Zn	<10	F	≤3
Lu	<0.3	Sn	<1	Cu	30	B	<0.03
Yb	<0.5	In*	<10	Ni	<10	Be	2
Tm	<0.3	Cd	<30	Co	10	Li	20
Er	<0.5	Ag*	<3	Fe	~5%		
Ho	<0.3	Pd	<3	Mn	100		

*Memory from previous sample.

TABLE 10. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
GROVE LIMESTONE
(ppmw)

U	<0.5	Dy	<0.2	Rh	<0.3	Cr	5
Th	<0.2	Tb	<0.3	Ru	<1	V	10
Bi	<0.1	Gd	<0.3	Mo	<3	Ti	2
Pb	3	Eu	<0.3	Nb	0.3	Sc	1
Tl	<0.1	Sm	<0.3	Zr	3	Ca	~30%
Hg	<0.3	Nd	0.5	Y	0.5	K	~0.3%
Au	<0.1	Pr	0.3	Sr	500	Cl	50
Pt	<0.3	Ce	2	Rb	5	S	50
Ir	<0.2	La	2	Br	0.5	P	10
Os	<0.2	Ba	100	Se*	<1	Si	~1%
Re	<0.2	Cs	<0.3	As	<0.5	Al	~0.1%
W	<0.3	I	1	Ge	<0.2	Mg	~1%
Ta	<0.3	Te	<0.3	Ga*	<0.5	Na	200
Hf	<0.5	Sb	<0.1	Zn	<3	F	≤10
Lu	<0.05	Sn	<1	Cu	2	B	1
Yb	<0.2	In*	<3	Ni	<2	Be	<0.005
Tm	<0.05	Cd	<3	Co	0.3	Li	3
Er	<0.2	Ag*	<1	Fe	~0.0200%		
Ho	<0.05	Pd	<2	Mn	100		

*Memory from previous sample.

TABLE 11. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
BED MATERIAL
(ppmw)

U	0.5	Dy	<0.2	Rh	<0.3	Cr	10
Th	0.2	Tb	<0.1	Ru	<1	V	10
Bi	<0.1	Gd	<0.3	Mo	<3	Ti	2
Pb	1	Eu	<0.3	Nb	0.5	Sc	1
Tl	<0.2	Sm	<0.5	Zr	20	Ca	~30%
Hg	<0.3	Nd	0.5	Y	3	K	~0.2%
Au	<0.1	Pr	0.3	Sr	500	Cl	50
Pt	<0.3	Ce	5	Rb	5	S	3,000
Ir	<0.2	La	2	Br	1	P	20
Os	<0.2	Ba	200	Se*	<2	Si	~0.5
Re	<0.2	Cs	0.3	As	1	Al	~0.1%
W	<0.3	I	0.5	Ge	10	Mg	2,000
Ta	<0.3	Te	<0.3	Ga*	<0.5	Na	200
Hf	<0.5	Sb	<0.2	Zn	<3	F	≤3
Lu	<0.05	Sn	<1	Cu	3	B	5
Yb	<0.2	In*	<2	Ni	<2	Be	<0.005
Tm	<0.1	Cd	<10	Co	0.3	Li	1
Er	<0.2	Ag*	<2	Fe	~0.1%		
Ho	<0.05	Pd	<2	Mn	20		

*Memory from previous sample.

TABLE 12. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
ASH
(ppmw)

U	5	Dy	2	Rh	<2	Cr	100
Th	10	Tb	0.5	Ru	<1	V	100
Bi	1	Gd	5	Mo	30	Ti	100
Pb	300	Eu	2	Nb	5	Sc	10
Tl	5	Sm	5	Zr	200	Ca	~30%
Hg	<0.3	Nd	5	Y	30	K	~2%
Au	<0.1	Pr	5	Sr	300	Cl	~1%
Pt	<0.3	Ce	50	Rb	20	S	~2%
Ir	<0.2	La	20	Br	20	P	50
Os	<0.2	Ba	300	Se*	<5	Si	~8%
Re	<0.2	Cs	5	As	10	Al	~1%
W	1	I	3	Ge	10	Mg	~2%
Ta	<0.3	Te	<0.3	Ga*	<5	Na	~1%
Hf	1	Sb	2	Zn	30	F	≤10
Lu	<0.1	Sn	200	Cu	2,000	B	30
Yb	0.2	In*	<3	Ni	50	Be	0.5
Tm	0.2	Cd	<10	Co	5	Li	10
Er	2	Ag*	<5	Fe	~1%		
Ho	0.5	Pd	<2	Mn	1,000		

*Memory from previous sample.

TABLE 13. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
PARTICULATES >27 μm
(ppmw)

U	2	Dy	3	Rh	<0.2	Cr	100
Th	5	Tb	0.5	Ru	<0.5	V	500
Bi	<0.1	Gd	1	Mo	20	Ti	1,000
Pb	20	Eu	1	Nb	3	Sc	30
Tl	5	Sm	5	Zr	200	Ca	~7%
Hg	<0.3	Nd	20	Y	30	K	~1%
Au	<0.1	Pr	5	Sr	200	Cl	800
Pt	<0.3	Ce	50	Rb	5	S	1,000
Ir	<0.2	La	50	Br	0.5	P	50
Os	<0.2	Ba	500	Se*	<5	Si	~20%
Re	<0.2	Cs	0.5	As	1	Al	~5%
W	0.3	I	0.5	Ge	3	Mg	5,000
Ta	<0.2	Te	<0.3	Ga*	<5	Na	3,000
Hf	2	Sb	0.2	Zn	<3	F	<5
Lu	<0.1	Sn	<2	Cu	50	B	50
Yb	1	In*	<1	Ni	200	Be	0.2
Tm	0.3	Cd	<3	Co	10	Li	10
Er	0.5	Ag*	<1	Fe	~2%		
Ho	0.5	Pd	<2	Mn	200		

*Memory from previous sample.

TABLE 14. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
PARTICULATES <27 μm
(ppmw)

U	2	Dy	2	Rh	<0.5	Cr	100
Th	5	Tb	0.3	Ru	<0.5	V	1,000
Bi	1	Gd	3	Mo	10	Ti	~1%
Pb	100	Eu	1	Nb	5	Sc	30
Tl	10	Sm	3	Zr	200	Ca	~7%
Hg	<0.3	Nd	20	Y	200	K	~2%
Au	<0.1	Pr	5	Sr	300	Cl	~1%
Pt	<0.3	Ce	20	Rb	10	S	3,000
Ir	<0.2	La	30	Br	1	P	200
Os	<0.2	Ba	300	Se*	<5	Si	~20%
Re	<0.2	Cs	2	As	3	Al	~5%
W	<0.3	I	5	Ge	10	Mg	~1%
Ta	<0.2	Te	<0.5	Ga*	<10	Na	5,000
Hf	<0.5	Sb	1	Zn	<3	F	<20
Lu	<0.1	Sn	10	Cu	50	B	300
Yb	1	In*	<1	Ni	100	Be	2
Tm	0.1	Cd	<3	Co	10	Li	20
Er	1	Ag*	<1	Fe	~2%		
Ho	0.5	Pd	<2	Mn	500		

*Memory from previous sample.

TABLE 15. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
SLUDGE
(ppmw)

U	20	Dy	10	Rh	<0.5	Cr	100
Th	50	Tb	2	Ru	<1	V	500
Bi	10	Gd	20	Mo	10	Ti	5,000
Pb	200	Eu	10	Nb	20	Sc	30
Tl	10	Sm	30	Zr	1,000	Ca	~7%
Hg	<0.3	Nd	50	Y	200	K.	~5%
Au	<0.1	Pr	20	Sr	500	Cl	200
Pt	<0.3	Ce	500	Rb	10	S	1,000
Ir	<0.2	La	200	Br	1	P	50
Os	<0.2	Ba	5,000	Se*	<5	Si	~20%
Re	<0.2	Cs	3	As	3	Al	~3%
W	2	I	1	Ge	20	Mg	5,000
Ta	<1	Te	<0.3	Ga*	<10	Na	3,000
Hf	10	Sb	1	Zn	<3	F	<5
Lu	0.5	Sn	100	Cu	50	B	50
Yb	5	In*	<1	Ni	200	Be	0.2
Tm	1	Cd	<3	Co	5	Li	10
Er	5	Ag*	<2	Fe	~5%		
Ho	3	Pd	<2	Mn	200		

*Memory from previous sample.

TABLE 16. SPARK SOURCE MASS SPECTROSCOPY--RUN 2
DETECTION LIMITS
(ppmw)

U	0.1	Dy	0.2	Rh	0.3	Cr	0.3
Th	0.1	Tb	0.1	Ru	0.3	V	0.3
Bi	0.1	Gd	0.3	Mo	0.3	Ti	0.3
Pb	0.2	Eu	0.3	Nb	0.3	Sc	0.3
Tl	0.1	Sm	0.3	Zr	0.3	Ca	0.5
Hg	0.3	Nd	0.5	Y	0.3	K	0.5
Au	0.1	Pr	0.2	Sr	0.3	Cl	0.5
Pt	0.3	Ce	0.5	Rb	0.3	S	0.5
Ir	0.2	La	0.3	Br	0.3	P	0.3
Os	0.2	Ba	0.5	Se	1.0	Si	2.0
Re	0.2	Cs	0.3	As	0.3	Al	10.0
W	0.3	I	0.3	Ge	0.2	Mg	3.0
Ta	0.2	Te	0.3	Ga	0.3	Na	0.1
Hf	0.3	Sb	0.1	Zn	1.0	F	0.5
Lu	0.05	Sn	1.0	Cu	0.3	B	0.03
Yb	0.2	In	1.0	Ni	2.0	Be	0.005
Tm	0.05	Cd	3.0	Co	0.3	Li	0.005
Er	0.2	Ag	1.0	Fe	0.3		
Ho	0.05	Pd	0.3	Mn	0.3		

TABLE 17. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS

Sample	Hg	Sb	As
Flue gas stream			
Run 1* (ppmw)	<MDL	N.R.	<50
Run 2 (ppmw)	0.007	N.R.	0.03
(ng/m^3)	9		39

*Analysis by OES. All components were near or below minimum detection limit (MDL not reported).

MDL = minimum detection limit.

N.R. = not reported.

TABLE 18. GAS CHROMATOGRAPHY* FOR INORGANIC GASES

Sample	SO_2 ppm	CO ppm	O_2%	CO_2%	THC ppmC
Run 1	700	790	6.5	14.3	85
Run 2	730	2,090	3.6	17.3	360
Run 3	620	4,790	3.3	18.3	900

*SO_2/CO/CO_2 by NDIR.
O_2 by EC - Polarograph.
THC by FID.

TABLE 19. CHEMILUMINESCENCE FOR NO_x FLUE GAS

Sample	NO_x concentrations (average) ppm
Run 1	415
Run 2	350
Run 3	300

TABLE 20. ANION ANALYSES*
(WT. PERCENT)

Sample	Cl	F	SO_4	SO_3	NO_3	NO_2	CO_3
Run 1							
Illinois #6 coal	†	†			†		0.68
Grove limestone	†	†			†		57.7
Particulates >27 μm	0.12	0.005			0.008		†
Particulates >27 μm	0.48	0.019			0.003		†
Run 2							
Illinois #6 coal			†	†	†	†	0.11
Limestone			†	†	<0.0003	<0.0003	57.6
Overflow bed material			20.2	0.04	0.0012	0.0007	†
Bed material-ash			16.4	0.16	0.012	0.0043	†
Particulates >27 μm			3.37	0.31	0.006	0.0008	6.45
Particulates <27 μm			5.34	<0.1	0.002	0.0006	5.97

*Analytical techniques and minimum detection limits for run 2 (run 1 MDL not reported): SO_4 by gravimetric, with 0.05 MDL; SO_3/CO_3 by titration, with 0.05 MDL; NO_3/NO_2 by colorimetric, with 0.0003 MDL; Cl and F MDL not reported.

†Below MDL.

No data indicates not reported in original document.

TABLE 21. LC FRACTIONATION--RUN 1

Particulates >27 µm				Particulates <27 µm				
Fraction	TCO	Grav µg/g	Total	Total	TCO	Grav µg/g	Total	Total
LC 1		309				750		
LC 2		10.9				35		
LC 3		21.8				55		
LC 4		29.1				85		
LC 5		4.7				50		
LC 6		9.6				75		
LC 7		6.4				35		
LC 8		13.1				20		

TABLE 22. LC FRACTIONATION--RUN 2

Bed Material*					Ash†				Sludge‡			
Fraction	TCO	Grav µg/m^3	Total	Total	TCO	Grav µg/m^3	Total	Total	TCO	Grav µg/m^3	Total	Total
LC 1		1,288				33.5				247		
LC 2		41				2.5				231		
LC 3		275				6.3				214		
LC 4		170				5.6				184		
LC 5		135				3.0				91.9		
LC 6		381				2.3				150		
LC 7		381				7.2				124		
LC 8		52.1				3.3				28.7		

*Multiply results of Bed Material by .023 to get µg/g.
†Multiply results of Ash by 11.7 to get µg/g.
‡Multiply results of Sludge by 1.42 to get µg/g.

TABLE 23. LC FRACTIONATION--RUN 2

	Particulates >27 µm*				Particulates <27 µm†				Flue gas			
Fraction	TCO	Grav µg/m^3	Total	Total	TCO	Grav µg/m^3	Total	Total	TCO	Grav µg/m^3	Total	Total
LC 1		372				144				4,158		
LC 2		422				83.2				605		
LC 3		144				150				2,364		
LC 4		112				118				1,873		
LC 5		73.8				70.7				610		
LC 6		66.8				141				919		
LC 7		162				536				1,033		
LC 8		38.7				8.1				10.9		

*Multiply results of particulates >27 µm by 0.06 to get µg/g.
†Multiply results of particulates <27 µm by 0.53 to get µg/g.

TABLE 24. IR REPORT
SAMPLES: FROM RUN 1

LC	Particulates >27 µm	Particulates <27 µm
1	Vinyl unsaturated hydrocarbons	Aliphatic hydrocarbons, vinyl unsaturated hydrocarbons.
2	Aliphatic esters	---
3	Aliphatic esters, ketone	Aliphatic esters
4	Phthalate ester	Conjugated ketone or quinone
5	---	Phthalate ester
6	---	---
7	---	---
8	---	---

TABLE 25. IR REPORT
SAMPLES: FROM RUN 2

LC	Bed Material	Ash
1	Aliphatic hydrocarbon containing a significant amount of vinyl unsaturation.	Aliphatic hydrocarbon containing a small amount of unsaturation including vinyl.
2	Contains only traces of hydrocarbon structure. Quantity of material is very low.	Aliphatic hydrocarbon containing a small amount of carbonyl.
3	Contains only traces of hydrocarbon structure. Quantity of material is very low.	Aliphatic hydrocarbon containing some aliphatic ester.
4	Contains only traces of hydrocarbon structure. Quantity of material is very low.	Carboxylic acid ester plus aliphatic ether groups, possibly a vinyl ether.
5	Contains only traces of hydrocarbon structure. Quantity of material is very low.	A small amount of phthalate ester.
6	Contains only traces of hydrocarbon structure. Quantity of material is very low.	---
7	Material concentration extremely low. A trace of aliphatic and carbonyl structure is present.	Primarily aliphatic--2 different carbonyls are present, one of which is probably a ketone.
8	(Not reported)	(Not reported)

TABLE 26. IR REPORT
SAMPLE: RUN 2 SLUDGE

LC 1	Aliphatic and fused ring aromatic hydrocarbons; pyrene and benzopyrene types are possible.
LC 2	Similar to 1 but concentration of fused ring aromatics is higher.
LC 3	Some of the fused ring aromatics of cuts #1 and #2 but primarily an aromatic ketone. Nitrile groups are present. A small amount of hydroxyl structure is present.
LC 4	Aromatic ketone and quinone structures. Small amounts of nitrile and hydroxyl.
LC 5	Aromatic ketone and quinone structures. A trace of nitrile. A small amount of anhydride or other strained ring carbonyl is probable.
LC 6	A complex mixture of many types of carbonyl, aliphatic, aromatic structures and with a trace of nitrile.
LC 7	Same as cut 6
LC 8	Only a trace of material; complex carbonyl structures.

TABLE 27. IR REPORT
SAMPLE: RUN 2 PARTICULATES

LC	>27 µm	<27 µm
1	Aliphatic hydrocarbons	Aliphatic and fused ring aromatics plus silicone.
2	Ester (very small amount of material).	Aliphatic and fused ring aromatics plus a small amount of ester.
3	Trace of ester plus other carbonyl.	A mixture of aliphatic and aromatic esters plus a trace of nitrile.
4	Trace of material containing several carbonyls.	Ester, ketone, and quinone are probable; both aromatic and aliphatic structure are present.
5	Trace of material containing carbonyls.	Aliphatic ester, probably unsaturated.
6	Trace of material containing carbonyl.	Aromatic strained ring or halogenated carbonyl.
7	Trace of material containing carbonyl.	(Not reported.)
8	---	(Not reported.)

STUDY NUMBER 13

DATA SOURCE:

EMISSIONS ASSESSMENT OF CONVENTIONAL COMBUSTION SYSTEMS

VOLUME I:

GAS-FIRED AND OIL-FIRED RESIDENTIAL HEATING SYSTEM SOURCE CATEGORIES

GCA-TR-77-30-G(1a)

DATA STATUS: Draft Report, September 1977

APPROVED BY: CONTRACTORS:

B. J. Matthews
TRW, Inc.
Energy Systems Group
One Space Park
Redondo Beach, California 90278
Contract No. 68-02-2197

N. F. Surprenant
GCA Corporation
GCA/Technology Division
Burlington Road
Bedford, Massachusetts 01730

PROJECT OFFICER:

Ronald A. Venezia
Chemical Processes Branch
Industrial Environmental Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

The first of a series of five environmental assessments, this study presents procedures used and results obtained in an evaluation of five gas fired and five oil-fired residential heating systems. Emission rates of particulates, gases, and trace elements were determined and added to an existing data base. Interpretation of these data conclude these emission sources are "not significant emitters of hazardous emissions," hence Level II analysis is not applied to the sources. This conclusion was based on the fact that the ratio of maximum ground level concentrations to predetermined hazardous levels was well below 0.1. Polycyclic organic material (POM) was also low with no detection of benzo(a)pyrene or dibenzo-anthracene.

The choice of sites to sample was based on their representation of other heaters currently in use across the country. Gas-fired units tested were warm air furnaces, whereas the oil-fired units were warm air as well as forced hot water types. All units used high pressure burners. The ten furnaces studied are identified as follows:

1. No. 100; gas, home unit; Gaffers and Sattler; No. 580; forced air furnace; 14 years old.
2. No. 101; gas, home unit; Gaffers and Sattler; No. 580; forced air furnace; 8 years old.
3. No. 102; gas, home unit, D&N Model C-80V; forced air furnace; 12 years old.
4. No. 103; gas, home unit; Sears Homart; forced air furnace; 20 years old.
5. No. 104; gas, home unit; Sears 735; forced air furnace; 6 years old.
6. No. 300; oil, FHW; American Standard; Model MF-A; 27 years old.
7. No. 301; oil, FHW; American Standard; No. W0391; 25 years old.
8. No. 302; oil, warm air; General Electric; No. 5KI + 39Cgl.2-g; ME1 burner; 21 years old.
9. No. 303; oil, warm air; U-Lubs; No. L862645; 5 years old.
10. No. 304; oil, warm air; Embassy Steel Products; 16 years old.

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GASEOUS GRAB

Collection of gas-fired furnace gases was by Tedlar bags using a stainless steel probe. Analysis for C_1-C_6 was by GC/FID; inorganic gases were separated by GC with thermal conductivity detection and quantified with use of standard gas mixtures. NO_x concentrations were determined electrochemically at gas-fired sites with a Theta sensor. At oil-fired sites, an evacuated flask was used for gas collection with analysis for NO_x using EPA Method 7. No data for this analysis were reported in the document.

SASS

The conventional SASS train was altered, due to expected particulates being less than 1 to 2 μm, by replacing the cyclones with a straight pipe leading to a Type N glass fiber filter in a heated oven. Organics were collected by a XAD-2 resin module and extracted with CH_2Cl_2 instead of pentane. Sampling occurred during the operational cycle of the furnace in which a typical cycle was 50 minutes on and 10 minutes off. Sampling times at gas-fired units was about 3 hours in which 30 m^3 of gas were sampled, while at oil-fired units 90 m^3 of gas were sampled over a period of 10 hours.

Particulates were prepared for SSMS by aqua regia extraction as opposed to the Level 1 preparation by Parr bomb.

FUGITIVE EMISSIONS

No sampling was performed in this study.

LIQUIDS AND SLURRIES

The oil-fired furnace fuel was analyzed for trace elements by SSMS.

No other samples of this nature were taken.

SOLIDS

No sampling was performed in this study.

Level 1 methods were used throughout this study, however, deviations from these methods did occur and are documented in the following list as well as in the text of this summary.

1. A HNO_3 module rinse for sample collection was not specified in Level 1 procedures. There were several references to a HNO_3 rinse in the GCA-TRW discussions of analytical methods.

2. Stainless steel components of the SASS train were rinsed with CH_2Cl_2 or a 1:1 (v/v) solution of CH_2Cl_2 and CH_3OH. Level 1 procedures specify only the 1:1 solution of CH_2Cl_2 and CH_3OH.

3. Solvent fraction #8 should have been eluted with 10 ml rather than 25 ml to comply with Level 1 procedures.

4. LC fractionation was not performed on the SASS train rinses.

5. LRMS analysis was omitted on LC fractions. Only TCO and IR were run.

6. AAS analysis of oil-fired emissions for toxic elements Hg, Sb, and As were omitted.

Organic analysis strategy used in this study is outlined in Figure 1.

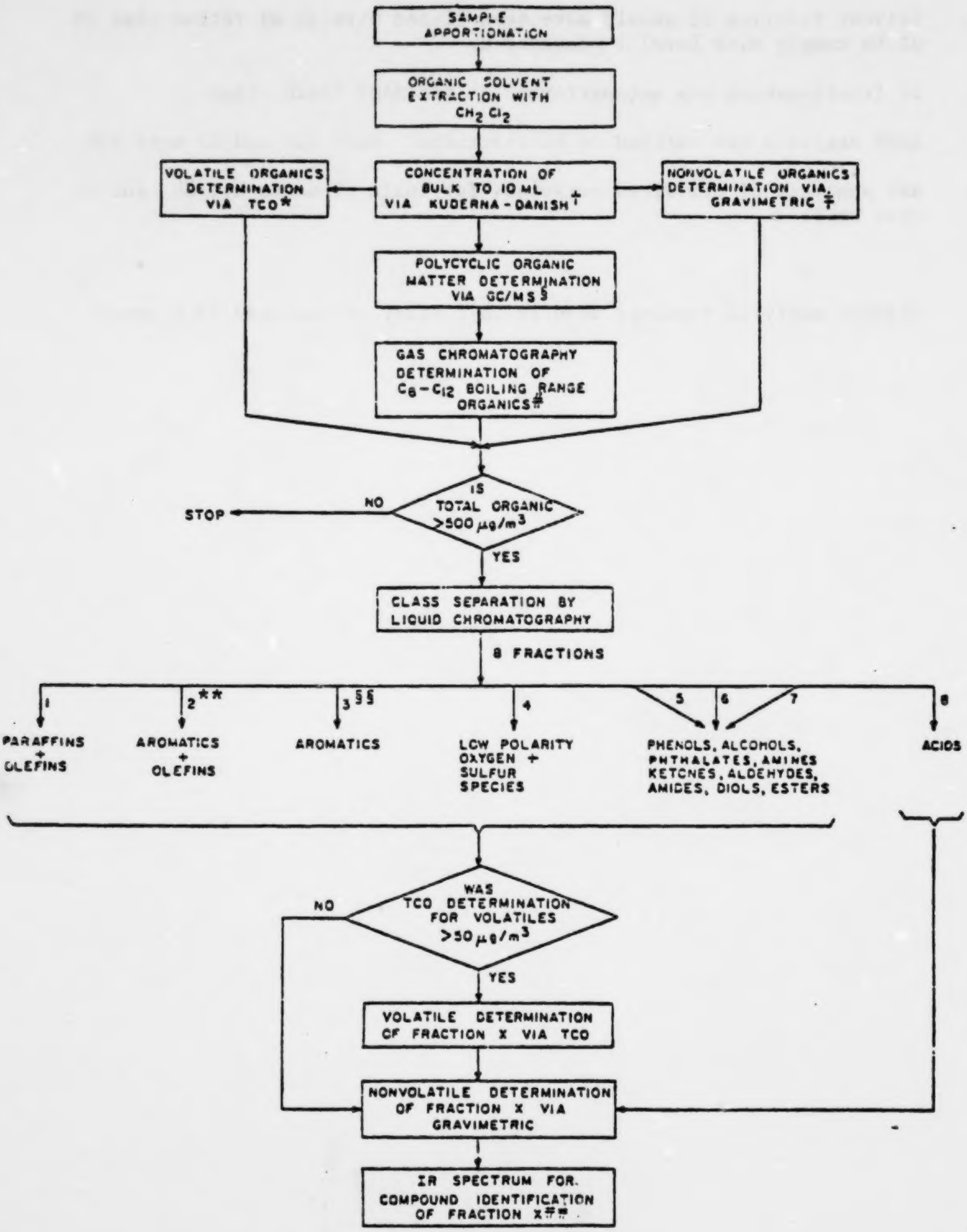
SAMPLE APPORTIONATION
ORGANIC SOLVENT EXTRACTION WITH CH_2Cl_2
VOLATILE ORGANICS DETERMINATION VIA TCO*
CONCENTRATION OF BULK TO 10 ML VIA KUDERNA-DANISH†
NONVOLATILE ORGANICS DETERMINATION VIA GRAVIMETRIC‡
POLYCYCLIC ORGANIC MATTER DETERMINATION VIA GC/MS§
GAS CHROMATOGRAPHY DETERMINATION OF C_8-C_{12} BOILING RANGE ORGANICS#
IS TOTAL ORGANIC >500 μg/m³
NO
STOP
YES
CLASS SEPARATION BY LIQUID CHROMATOGRAPHY
8 FRACTIONS
1
2**
3§§
4
5
6
7
8
PARAFFINS + OLEFINS
AROMATICS + OLEFINS
AROMATICS
LOW POLARITY OXYGEN + SULFUR SPECIES
PHENOLS, ALCOHOLS, PHTHALATES, AMINES KETONES, ALDEHYDES, AMIDES, DIOLS, ESTERS
ACIDS
WAS TCO DETERMINATION FOR VOLATILES >50 μg/m³
NO
YES
VOLATILE DETERMINATION OF FRACTION X VIA TCO
NONVOLATILE DETERMINATION OF FRACTION X VIA GRAVIMETRIC
IR SPECTRUM FOR COMPOUND IDENTIFICATION OF FRACTION X‡‡

Figure 1. Level 1 organic analysis strategy.

*TCO is a gas chromatographic technique for detecting hydrocarbon-equivalent compounds boiling between 90° and 290° C.

†Kuderna-Danish concentration is a glass apparatus for evaporating bulk amounts of solvents.

‡Gravimetric determination tare weighs organic compounds boiling above 290° C.

§GC/MS is sequential gas chromatography mass spectrometry which separates a mixture of compounds and identifies each.

#Boiling range determination is a gas chromatographic technique designed to quantitate hydrocarbon-equivalent compounds boiling in the ranges, 110° to 140° C, 140° to 160° C, 160° to 180° C, 180° to 200° C, and 200° to 220° C.

**This fraction is composed mostly of halo-aromatics; e.g., PCB.

§§This fraction is composed mostly of polycyclic organic aromatics.

##Infrared spectroscopy identifies functional groups present in organics; e.g., alcohol, amine, carboxy, aromatic, etc.

Figure 1 (con.)

TABLE 1. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 100
SASS PARTICULATE FILTER*
(μg)

U	<0.1	Dy	<0.05	Rh	<0.2	Cr	<1
Th	<0.1	Tb	<0.05	Ru	<0.1	V	<0.1
Bi	<0.1	Gd	<0.1	Mo	<0.1	Ti	<5
Pb	<15	Eu	<0.05	Nb	<0.05	Sc	<0.05
Tl	<0.1	Sm	<0.1	Zr	<0.1	Ca	<50
Hg		Nd	<0.1	Y	<0.05	K	<30
Au	<0.05	Pr	<0.05	Sr	<1	Cl	
Pt		Ce	<0.1	Rb	<1	S	<200
Ir	<0.1	La	<0.05	Br	<0.1	P	<2
Os	<0.1	Ba	≤10	Se	<1	Si	
Re	<0.1	Cs	<1.0	As	<1	Al	<500
W	<0.1	I	<0.05	Ge	<0.05	Mg	15
Ta		Te	<0.1	Ga	<0.05	Na	<1,500
Hf	<0.1	Sb	<2	Zn	200	F	<50
Lu	<0.05	Sn	<2	Cu	<50	B	<3
Yb	<0.01	In		Ni	50	Be	<0.05
Tm	<0.05	Cd	<0.1	Co	<0.005	Li	0.1
Er	<0.05	Ag		Fe	<50		
Ho	<0.05	Pd	<0.1	Mn	<1		

*Aqua regia digestion; no Parr bomb.

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 100
XAD-2 RESIN
(μg)

U	<8	Dy	<30	Rh	<30	Cr	120
Th	<8	Tb	<8	Ru	<15	V	<8
Bi	<8	Gd	<30	Mo	<40	Ti	30
Pb	<300	Eu	<8	Nb	<8	Sc	<8
Tl	<15	Sm	<15	Zr	<8	Ca	<3,500
Hg		Nd	<15	Y	<8	K	<700
Au	<8	Pr	<8	Sr	45	Cl	
Pt		Ce	<15	Rb	<15	S	<9,000
Ir	<15	La	<8	Br	<8	P	<350
Os	<15	Ba	<300	Se	15	Si	
Re	<15	Cs	<8	As	<30	Al	<250
W	<15	I	<8	Ge	<8	Mg	<25
Ta		Te	<15	Ga	<8	Na	<20,000
Hf	<15	Sb	60	Zn	<500	F	<400
Lu	<8	Sn	<500	Cu	<10,000	B	300
Yb	<15	In		Ni	<1,000	Be	<4
Tm	<8	Cd	<8	Co	<8	Li	<8
Er	<15	Ag		Fe	1,500		
Ho	<8	Pd	<15	Mn	60		

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 100
IMPINGER, MODULE RINSE, CONDENSATE
(µg)

U	<3	Dy	<9	Rh	<9	Cr	<60
Th	<3	Tb	<3	Ru	<6	V	<3
Bi	<3	Gd	<9	Mo	<15	Ti	<15
Pb	<70	Eu	<3	Nb	<3	Sc	<3
Tl	<6	Sm	<6	Zr	<3	Ca	<300
Hg		Nd	<6	Y	<3	K	<80
Au	<3	Pr	<3	Sr	<3	Cl	
Pt		Ce	<6	Rb	<6	S	<10,000
Ir	<6	La	<3	Br	<3	P	<20
Os	<6	Ba	41	Se	<3	Si	
Re	<6	Cs	<3	As	<3	Al	<250
W	<6	I	<3	Ge	<3	Mg	<15
Ta		Te	<6	Ga	<3	Na	<20,000
Hf	<6	Sb	<3	Zn	<200	F	<150
Lu	<3	Sn	<130	Cu	<30	B	<3
Yb	<6	In		Ni	<90	Be	<1.5
Tm	<3	Cd	<6	Co	<3	Li	<3
Er	<6	Ag		Fe	<300		
Ho	<3	Pd	<6	Mn	<15		

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 100
TOTAL EMISSIONS FOUND
($\mu g/m^3$)

U	<0.3	Dy	<1.5	Rh	<1.5	Cr	3-6
Th	<0.3	Tb	<0.3	Ru	<0.6	V	<0.3
Bi	<0.3	Gd	<1.5	Mo	<2	Ti	1-2
Pb	<15	Eu	<0.3	Nb	<0.3	Sc	<0.3
Tl	<0.6	Sm	<0.6	Zr	<0.3	Ca	<150
Hg		Nd	<0.6	Y	<0.3	K	<30
Au	<0.3	Pr	<0.3	Sr	<2	Cl	
Pt		Ce	<0.6	Rb	<0.6	S	<600
Ir	<0.6	La	<0.3	Br	<0.3	P	<15
Os	<0.6	Ba	1-20	Se	0.5	Si	
Re	<0.6	Cs	0.03-0.3	As	<1	Al	<60
W	<0.6	I	<0.3	Ge	<0.3	Mg	0.5-1.5
Ta		Te	<0.6	Ga	<0.3	Na	<1,500
Hf	<0.6	Sb	1.8	Zn	6-30	F	<20
Lu	<0.3	Sn	<20	Cu	<300	B	<10
Yb	<0.6	In		Ni	2-30	Be	<0.2
Tm	<0.3	Cd	<0.5	Co	<0.3	Li	<0.3
Er	<0.6	Ag		Fe	50-70		
Ho	<0.3	Pd	<0.6	Mn	<2		

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 101
XAD-2 RESIN
(μg)

U	<8	Dy	<30	Rh	<30	Cr	<200
Th	<8	Tb	<8	Ru	<15	V	<8
Bi	<8	Gd	<30	Mo	<60	Ti	23
Pb	<400	Eu	<8	Nb	<8	Sc	<8
Tl	<15	Sm	<15	Zr	<8	Ca	<4,000
Hg		Nd	<15	Y	<8	K	<500
Au	<8	Pr	<8	Sr	<30	Cl	
Pt		Ce	<15	Rb	<30	S	<8,000
Ir	<15	La	<8	Br	<8	P	<200
Os	<15	Ba	<300	Se	<30	Si	
Re	<15	Cs	<8	As	<20	Al	<1,000
W	<15	I	<8	Ge	<8	Mg	<200
Ta		Te	<8	Ga	<8	Na	<11,000
Hf	<15	Sb	15	Zn	<300	F	<800
Lu	<8	Sn	<10	Cu	<10,000	B	15
Yb	<15	In		Ni	<300	Be	<8
Tm	<8	Cd	<30	Co	<8	Li	<8
Er	<15	Ag		Fe	<800		
Ho	<8	Pd	<15	Mn	<20		

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 101
IMPINGER, MODULE RINSE, CONDENSATE
(μg)

U	<5	Dy	<15	Rh	<15	Cr	<700
Th	<5	Tb	<5	Ru	<9	V	<0.9
Bi	<5	Gd	<15	Mo	<60	Ti	<9
Pb	<200	Eu	<5	Nb	<5	Sc	<5
Tl	<9	Sm	<9	Zr	<5	Ca	<2,000
Hg		Nd	<9	Y	<5	K	3,600
Au	<5	Pr	<5	Sr	<20	Cl	
Pt		Ce	<9	Rb	<20	S	<400,000
Ir	<9	La	<5	Br	<9	P	<200
Os	<9	Ba	40	Se	<5	Si	
Re	<9	Cs	<5	As	45	Al	450
W	<9	I	<5	Ge	<5	Mg	<300
Ta		Te	<9	Ga	<5	Na	36,000
Hf	<9	Sb	<30	Zn	<5,000	F	1,600
Lu	<5	Sn	<400	Cu	<1,000	B	<20
Yb	<9	In		Ni	<2,000	Be	<2
Tm	<5	Cd	<300	Co	50	Li	<10
Er	<9	Ag		Fe	<2,000		
Ho	<5	Pd	<9	Mn	130		

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 101
TOTAL EMISSIONS FOUND
($\mu g/m^3$)

U	<0.5	Dy	<2	Rh	<2	Cr	<40
Th	<0.5	Tb	<0.5	Ru	<0.8	V	<0.8
Bi	<0.5	Gd	<2	Mo	<4	Ti	0.77
Pb	<20	Eu	<0.5	Nb	<0.5	Sc	<0.5
Tl	<0.8	Sm	<0.8	Zr	<0.5	Ca	<300
Hg		Nd	<0.8	Y	<0.5	K	120
Au	<0.5	Pr	<0.5	Sr	<2	Cl	
Pt		Ce	<0.8	Rb	<2	S	<20,000
Ir	<0.8	La	<0.5	Br	<0.8	P	<20
Os	<0.8	Ba	<1-20	Se	<2	Si	
Re	<0.8	Cs	<0.5	As	1.5	Al	15-50
W	<0.8	I	<0.5	Ge	<0.5	Mg	<20
Ta		Te	<0.8	Ga	<0.5	Na	1,200-2,000
Hf	<0.8	Sb	0.5-1.5	Zn	<200	F	50-300
Lu	<0.5	Sn	<20	Cu	<400	B	0.5-2
Yb	<0.8	In		Ni	<80	Be	<0.4
Tm	<0.5	Cd	<10	Co	2	Li	<0.6
Er	<0.8	Ag		Fe	<100		
Ho	<0.5	Pd	<0.8	Mn	<4		

TABLE 8. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 102
XAD-2 RESIN
(μg)

U	<8	Dy	<25	Rh	<25	Cr	400
Th	<8	Tb	<8	Ru	<15	V	<8
Bi	<8	Gd	<25	Mo	120	Ti	15
Pb	<400	Eu	<8	Nb	<25	Sc	<8
Tl	<15	Sm	<15	Zr	<8	Ca	1,300
Hg		Nd	<15	Y	<8	K	<900
Au	<8	Pr	<8	Sr	38	Cl	
Pt		Ce	<15	Rb	<20	S	<40,000
Ir	<15	La	<8	Br	<8	P	<1,400
Os	<15	Ba	300	Se	<15	Si	
Re	<15	Cs	<8	As	<70	Al	1,400
W	<15	I	<8	Ge	<8	Mg	<400
Ta		Te	<15	Ga	<8	Na	<15,000
Hf	<15	Sb	<15	Zn	<150	F	<800
Lu	<8	Sn	<200	Cu	<40,000	B	30
Yb	<15	In		Ni	<1,500	Be	<8
Tm	<8	Cd	53	Co	<8	Li	<20
Er	<15	Ag		Fe	750		
Ho	<8	Pd	<15	Mn	<40		

TABLE 9. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 102
IMPINGER, MODULE RINSE, CONDENSATE
(μg)

U	<3	Dy	<8	Rh	<8	Cr	<400
Th	<3	Tb	<3	Ru	<5	V	<3
Bi	<3	Gd	<8	Mo	<40	Ti	<30
Pb	<100	Eu	<3	Nb	<5	Sc	<3
Tl	<5	Sm	<5	Zr	<5	Ca	<1,000
Hg		Nd	<5	Y	<5	K	<500
Au	<3	Pr	<3	Sr	<15	Cl	
Pt		Ce	<5	Rb	<3	S	<10,000
Ir	<5	La	<3	Br	<3	P	<300
Os	<5	Ba	38	Se	4-0	Si	
Re	<5	Cs	<3	As	15	Al	<500
W	<5	I	<3	Ge	<3	Mg	<100
Ta		Te	<5	Ga	<3	Na	<18,000
Hf	<5	Sb	<15	Zn	<500	F	<200
Lu	<3	Sn	<100	Cu	<400	B	<3
Yb	<5	In		Ni	<400	Be	<3
Tm	<3	Cd	60	Co	<3	Li	<3
Er	<5	Ag		Fe	380		
Ho	<3	Pd	<5	Mn	48		

TABLE 10. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 102
TOTAL EMISSIONS FOUND
($\mu g/m^3$)

U	<0.3	Dy	<1	Rh	<1.5	Cr	10-20
Th	<0.3	Tb	<0.3	Ru	<1	V	<0.3
Bi	<0.3	Gd	<1.5	Mo	4	Ti	0.5-2
Pb	<20	Eu	<0.3	Nb	<1	Sc	<0.3
Tl	<0.6	Sm	<0.6	Zr	<0.3	Ca	40-80
Hg		Nd	<0.6	Y	<0.3	K	<50
Au	<0.3	Pr	<0.3	Sr	1.2	Cl	
Pt		Ce	<0.6	Rb	<0.6	S	<1,500
Ir	<0.6	La	<0.3	Br	<0.3	P	<20
Os	<0.6	Ba	10	Se	0.1-0.6	Si	
Re	<0.6	Cs	<0.3	As	0.5-3	Al	40-60
W	<0.6	I	<0.3	Ge	<0.3	Mg	<15
Ta		Te	<0.6	Ga	<0.3	Na	<1,000
Hf	<0.6	Sb	<1	Zn	<20	F	<30
Lu	<0.3	Sn	<10	Cu	<1,500	B	<1
Yb	<0.6	In		Ni	<60	Be	<0.3
Tm	<0.3	Cd	1-4	Co	<0.3	Li	<0.6
Er	<0.6	Ag		Fe	34		
Ho	<0.3	Pd	<0.6	Mn	1-3		

TABLE 11. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 103
XAD-2 RESIN
(μg)

U	<8	Dy	<23	Rh	<23	Cr	<100
Th	<8	Tb	<8	Ru	<15	V	<9
Bi	<8	Gd	<15	Mo	<12	Ti	<20
Pb	<300	Eu	<8	Nb	<8	Sc	<8
Tl	<15	Sm	<15	Zr	<3	Ca	<3,200
Hg		Nd		Y	<8	K	<700
Au	<8	Pr	<8	Sr	<20	Cl	
Pt		Ce	3	Rb	<11	S	<38K
Ir	<15	La	<8	Br	<7	P	<300
Os	<15	Ba	<140	Se	<8	Si	
Re	<15	Cs	2.3	As	<5	Al	<700
W	<15	I	<8	Ge	<8	Mg	<140
Ta		Te	<15	Ga	<8	Na	<5,000
Hf	<15	Sb	<10	Zn	<230	F	<8
Lu	<8	Sn	<230	Cu	<750	B	<6
Yb	<15	In		Ni	<1,100	Be	<2
Tm	<8	Cd	<10	Co	<2	Li	<4
Er	<15	Ag		Fe	<800		
Ho	<8	Pd	<15	Mn	<40		

TABLE 12. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 103
IMPINGER, MODULE RINSE, CONDENSATE
(μg)

U	<2	Dy	<6	Rh	<6	Cr	<200
Th	<2	Tb	<2	Ru	<4	V	<2
Bi	<2	Gd	<6	Mo	<20	Ti	<6
Pb	<50	Eu	<2	Nb	<4	Sc	<2
Tl	<4	Sm	<4	Zr	<2	Ca	<800
Hg		Nd	<4	Y	<2	K	<200
Au	<2	Pr	<2	Sr	<13	Cl	
Pt		Ce	1.8	Rb	<4	S	<3,000
Ir	<4	La	<2	Br	<2	P	<10
Os	<4	Ba	<2	Se	<2	Si	
Re	<4	Cs	20	As	<4	Al	<100
W	<15	I	<2	Ge	<2	Mg	<80
Ta		Te	<4	Ga	<2	Na	<4,000
Hf	<4	Sb	<2	Zn	<300	F	<2
Lu	<2	Sn	<100	Cu	<40	B	<2
Yb	<4	In		Ni	<200	Be	<2
Tm	<2	Cd	<20	Co	<2	Li	<2
Er	<4	Ag		Fe	<600		
Ho	<2	Pd	<4	Mn	31		

TABLE 13. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 103
TOTAL EMISSIONS FOUND
($\mu g/m^3$)

U	<0.3	Dy	<1	Rh	<1	Cr	<9
Th	<0.3	Tb	<0.3	Ru	<0.6	V	<0.3
Bi	<0.3	Gd	<0.6	Mo	<1	Ti	<0.8
Pb	<10	Eu	<0.3	Nb	<0.4	Sc	<0.3
Tl	<0.6	Sm	<0.6	Zr	<0.2	Ca	<100
Hg		Nd	<0.3	Y	<0.3	K	<30
Au	<0.3	Pr	<0.3	Sr	<1	Cl	
Pt		Ce	0.15	Rb	<0.5	S	<1,000
Ir	<0.6	La	<0.3	Br	<0.3	P	<9
Os	<0.6	Ba	<0.3	Se	<0.3	Si	
Re	<0.6	Cs	0.67	As	<0.3	Al	<30
W	<1	I	<0.3	Ge	<0.3	Mg	<6
Ta		Te	<0.6	Ga	<0.3	Na	<300
Hf	<0.6	Sb	<0.4	Zn	<15	F	<0.3
Lu	<0.3	Sn	<10	Cu	<30	B	<0.3
Yb	<0.6	In		Ni	<40	Be	<0.1
Tm	<0.3	Cd	<1	Co	<0.1	Li	<0.2
Er	<0.6	Ag		Fe	<40		
Ho	<0.3	Pd	<0.6	Mn	0.94-2		

TABLE 14. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 104
SASS PARTICULATE FILTER*
(μg)

U	<0.02	Dy	<0.04	Rh	<0.04	Cr	<0.39
Th	<0.02	Tb	<0.01	Ru	<0.02	V	<0.024
Bi	<0.08	Gd	<0.04	Mo	<0.057	Ti	<1.5
Pb	<3.3	Eu	<0.02	Nb	<0.01	Sc	<0.01
Tl	<0.04	Sm	<0.03	Zr	<0.63	Ca	<58
Hg		Nd	<0.06	Y	0.015	K	<17
Au	<0.02	Pr	<0.01	Sr	<0.18	Cl	
Pt		Ce	<0.025	Rb	<0.14	S	<320
Ir	<0.02	La	<0.015	Br	<0.056	P	<1.3
Os	<0.03	Ba	<2.3	Se	<0.46	Si	
Re	<0.02	Cs	<0.057	As	<0.079	Al	<370
W	<0.04	I	<0.01	Ge	<0.01	Mg	<8.3
Ta		Te	<0.02	Ga	<0.01	Na	<1,900
Hf	<0.03	Sb	<0.081	Zn	510	F	<3.4
Lu	<0.02	Sn	<0.32	Cu	<61	B	<10
Yb	<0.04	In		Ni	<88	Be	<0.0016
Tm	<0.02	Cd	<0.23	Co	<0.018	Li	<0.044
Er	<0.03	Ag		Fe	<9		
Ho	<0.02	Pd	<0.02	Mn	<0.23		

*Aqua regia digestion; no Parr bomb.

TABLE 15. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 104
XAD-2 RESIN
(μg)

U	<8	Dy	<15	Rh	<15	Cr	<20
Th	<8	Tb	<4	Ru	<8	V	<2
Bi	<8	Gd	<15	Mo	<5	Ti	<6
Pb	<130	Eu	<8	Nb	<4	Sc	<4
Tl	<8	Sm	<10	Zr	<4	Ca	<1,500
Hg		Nd	<20	Y	<4	K	<300
Au	<8	Pr	<4	Sr	<20	Cl	
Pt		Ce	<4	Rb	<4	S	<8,000
Ir	<8	La	<4	Br	<1.5	P	<200
Os	<10	Ba	<200	Se	<8	Si	
Re	<8	Cs	<4	As	<5	Al	5,700
W	<15	I	<4	Ge	<4	Mg	<90
Ta		Te	<8	Ga	<4	Na	<5,000
Hf	<10	Sb	<15	Zn	<100	F	<2
Lu	<8	Sn	<300	Cu	<500	B	<3
Yb	<15	In		Ni	<100	Be	<0.1
Tm	<15	Cd	<6	Co	<1	Li	<4
Er	<10	Ag		Fe	<500		
Ho	<8	Pd	<8	Mn	<10		

TABLE 16. SPARK SOURCE MASS SPECTROSCOPY
GAS FIRED FURNACE: SITE 104
IMPINGER, MODULE RINSE, CONDENSATE
(μg)

U	<2	Dy	<4	Rh	<4	Cr	<100
Th	<2	Tb	<1	Ru	<2	V	<1.5
Bi	<200	Gd	<4	Mo	<30	Ti	<0.2
Pb	75	Eu	<2	Nb	<1	Sc	<1
Tl	<2	Sm	<3	Zr	<1	Ca	<500
Hg		Nd	<6	Y	<1	K	<170
Au	<2	Pr	<1	Sr	<6	Cl	
Pt		Ce	<1	Rb	<3	S	7,600
Ir	<2	La	<1	Br	<1	P	560
Os	<3	Ba	340	Se	<2	Si	
Re	<2	Cs	5.4	As	<5	Al	<130
W	<4	I	<1	Ge	<1	Mg	<47
Ta		Te	<2	Ga	<1	Na	<6,000
Hf	<3	Sb	<9	Zn	<800	F	<0.9
Lu	<2	Sn	<6,000	Cu	<70	B	<0.7
Yb	<4	In		Ni	<170	Be	<0.1
Tm	<4	Cd	<50	Co	<2	Li	<0.5
Er	<3	Ag		Fe	<12,000		
Ho	<2	Pd	<2	Mn	<140		

TABLE 17. SPARK SOURCE MASS SPECTROSCOPY
GAS-FIRED FURNACE: SITE 104
TOTAL EMISSIONS FOUND
($\mu g/m^3$)

U	<0.3	Dy	<0.6	Rh	<0.6	Cr	<4
Th	<0.3	Tb	<0.2	Ru	<0.3	V	<0.1
Bi	<6	Gd	<0.6	Mo	<1	Ti	<0.2
Pb	2.3-6	Eu	<0.3	Nb	<0.2	Sc	<0.2
Tl	<0.3	Sm	<0.4	Zr	<0.2	Ca	<70
Hg		Nd	<1	Y	<0.2	K	<20
Au	<0.3	Pr	<0.2	Sr	<1	Cl	
Pt		Ce	<0.2	Rb	<0.2	S	240-470
Ir	<0.3	La	<0.2	Br	<0.1	P	17-24
Os	<0.4	Ba	10-15	Se	<0.3	Si	
Re	<0.3	Cs	0.17-0.3	As	<0.3	Al	180
W	<0.6	I	<0.2	Ge	<0.2	Mg	<4
Ta		Te	<0.3	Ga	<0.2	Na	<400
Hf	<0.4	Sb	<0.8	Zn	<30	F	<0.2
Lu	<0.3	Sn	<200	Cu	<20	B	<0.4
Yb	<0.6	In		Ni	<10	Be	<0.006
Tm	<0.6	Cd	<2	Co	<0.1	Li	<0.2
Er	<0.4	Ag		Fe	<400		
Ho	<0.3	Pd	<0.3	Mn	<5		

TABLE 18. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 300
FUEL OIL
(ppm)

U	<0.01	Dy	<0.03	Rh	<0.03	Cr	0.61
Th	<0.01	Tb	<0.01	Ru	<0.02	V	0.03
Bi	<0.02	Gd	<0.03	Mo	0.072	Ti	0.19
Pb	1.4	Eu	<0.01	Nb	<0.05	Sc	<0.01
Tl	<0.02	Sm	<0.02	Zr	<0.07	Ca	15
Hg		Nd	<0.05	Y	<0.01	K	2.6
Au	<0.01	Pr	<0.03	Sr	0.12	Cl	<25
Pt		Ce	0.12	Rb	0.03	S	300
Ir	<0.02	La	0.08	Br	<0.02	P	0.38
Os	<0.02	Ba	0.41	Se	<0.02	Si	
Re	<0.02	Cs	<0.01	As	<0.03	Al	7.0
W	<0.03	I	<0.01	Ge	<0.01	Mg	1.8
Ta		Te	<0.02	Ga	<0.01	Na	<30
Hf	<0.02	Sb	<0.03	Zn	1.7	F	<0.5
Lu	<0.01	Sn		Cu	1.3	B	<0.005
Yb	<0.02	In		Ni	6.0	Be	<0.005
Tm	<0.01	Cd	<0.01	Co	0.01	Li	0.02
Er	<0.02	Ag		Fe	4.5		
Ho	<0.01	Pd	<0.02	Mn	2.2		

TABLE 19. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 300
SASS PARTICULATE FILTER*
(μg)

U	<1	Dy	<3	Rh	<1	Cr	<5
Th	<1	Tb	<1	Ru	<1	V	<1
Bi	<3	Gd	<3	Mo	2.3	Ti	<16
Pb	1,200	Eu	<1	Nb	<1	Sc	<1
Tl	<3	Sm	<2	Zr	15	Ca	<320
Hg		Nd	<3	Y	<1	K	<140
Au	<1	Pr	<1	Sr	<2	Cl	
Pt		Ce	<2	Rb	<1	S	14,000
Ir	<1	La	<1	Br	<1	P	<11
Os	<2	Ba	<30	Se	<2	Si	
Re	<2	Cs	<1	As	<2	Al	<250
W	<3	I	<1	Ge	<1	Mg	<30
Ta		Te	<2	Ga	<1	Na	<500
Hf	<2	Sb	<4	Zn	380	F	<50
Lu	<1	Sn		Cu	73	B	<3
Yb	<2	In		Ni	<36	Be	<0.1
Tm	<1	Cd	20	Co	<1	Li	<0.1
Er	<2	Ag		Fe	<220		
Ho	<1	Pd	<1	Mn	<10		

*Aqua regia digestion; no Parr bomb.

TABLE 20. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 300
XAD-2 RESIN
(μg)

U	<1	Dy	<3	Rh	<3	Cr	20
Th	<1	Tb	<1	Ru	<2	V	3.2
Bi	<3	Gd	<3	Mo	5	Ti	17
Pb	<100	Eu	<1	Nb	<3	Sc	<1
Tl	<2	Sm	<2	Zr	<1	Ca	<60
Hg		Nd	<2	Y	<1	K	<50
Au	<1	Pr	<1	Sr	10	Cl	<500
Pt		Ce	<3	Rb	58	S	1,400
Ir	<2	La	<1	Br	<2	P	<20
Os	<2	Ba	81	Se	12	Si	
Re	<2	Cs	54	As	10	Al	380
W	<3	I	<1	Ge	<1	Mg	<120
Ta		Te	<2	Ga	<2	Na	<1,300
Hf	<2	Sb	5.4	Zn	<90	F	210
Lu	<1	Sn		Cu	<35	B	14
Yb	<2	In		Ni	36	Be	<0.3
Tm	<2	Cd	<2	Co	<1	Li	<0.2
Er	<1	Ag		Fe	315		
Ho	<1	Pd	<2	Mn	35		

TABLE 21. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 300
CONDENSATE/HNO_3 RINSE
(μg)

U	<1	Dy	<2	Rh	<1	Cr	290
Th	<1	Tb	<1	Ru	<1	V	<2
Bi	<1	Gd	<2	Mo	83	Ti	<25
Pb	<130	Eu	<1	Nb	<3	Sc	<12
Tl	<2	Sm	<2	Zr	<2	Ca	<110
Hg		Nd	<3	Y	<1	K	<20
Au	<1	Pr	<1	Sr	<3	Cl	<1,000
Pt		Ce	<2	Rb	<3	S	62,000
Ir	<1	La	<1	Br	<1	P	140
Os	<2	Ba	<18	Se	<12	Si	
Re	<1	Cs	1	As	<3	Al	370
W	25	I	<4	Ge	2	Mg	400
Ta		Te	<1	Ga	<1	Na	9,300
Hf	<2	Sb	<4.8	Zn	<55	F	<100
Lu	<1	Sn		Cu	<1,000	B	<1
Yb	<2	In		Ni	<1,000	Be	<0.1
Tm	<1	Cd	16	Co	<10	Li	<0.1
Er	<2	Ag		Fe	<1,300		
Ho	<1	Pd	<1	Mn	95		

TABLE 22. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 300
TOTAL EMISSIONS FOUND
(μg/m^3)

U	<0.04	Dy	<0.09	Rh	<0.06	Cr	3.4
Th	<0.04	Tb	<0.04	Ru	<0.05	V	<0.08
Bi	<0.05	Gd	<0.09	Mo	0.98	Ti	0.2-0.6
Pb	13	Eu	<0.04	Nb	<0.07	Sc	<0.2
Tl	<0.1	Sm	<0.07	Zr	0.16	Ca	<6
Hg		Nd	<0.09	Y	<0.04	K	0.2-3
Au	<0.04	Pr	<0.04	Sr	0.1-0.2	Cl	<0.5
Pt		Ce	<0.08	Rb	0.63	S	840
Ir	<0.08	La	<0.04	Br	<0.06	P	1.5
Os	<0.09	Ba	0.09-1.5	Se	0.1-0.4	Si	
Re	<0.06	Cs	0.60	As	0.1-0.2	Al	7-11
W	0.3	I	<0.07	Ge	<0.06	Mg	4-7
Ta		Te	<0.06	Ga	<0.06	Na	100
Hf	<0.08	Sb	0.06-0.2	Zn	4.4	F	<5
Lu	<0.04	Sn		Cu	0.7-15	B	0.15
Yb	<0.08	In		Ni	0.3-11	Be	<.005
Tm	<0.05	Cd	0.20-0.5	Co	<0.2	Li	<0.04
Er	<0.06	Ag		Fe	3-30		
Ho	<0.04	Pd	<0.05	Mn	1.4		

TABLE 23. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 301
FUEL OIL
(ppm)

U	<0.02	Dy	<0.03	Rh	<0.03	Cr	<0.3
Th	<0.02	Tb	<0.01	Ru	<0.02	V	<0.01
Bi	<0.02	Gd	<0.03	Mo	<0.05	Ti	<0.2
Pb	1.1	Eu	<0.01	Nb	<0.01	Sc	<0.01
Tl	<0.03	Sm	<0.02	Zr	<0.02	Ca	<4
Hg		Nd	<0.05	Y	<0.01	K	1.3
Au	<0.01	Pr	<0.01	Sr	<0.01	Cl	<10
Pt		Ce	0.10	Rb	<0.03	S	320
Ir	<0.02	La	<0.03	Br	<0.01	P	0.06
Os	<0.02	Ba	<0.6	Se		Si	
Re	<0.02	Cs	<0.01	As	<0.01	Al	<8
W	<0.03	I	<0.01	Ge	<0.01	Mg	<0.4
Ta		Te	<0.02	Ga	<0.01	Na	<20
Hf	<0.02	Sb	<0.02	Zn	<2	F	<0.01
Lu	<0.01	Sn	<0.4	Cu	<4	B	<0.01
Yb	<0.02	In		Ni	3.2	Be	<0.01
Tm	<0.01	Cd	0.15	Co	0.030	Li	<0.007
Er	<0.02	Ag		Fe	<7		
Ho	<0.01	Pd	<0.02	Mn	<0.2		

TABLE 24. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 301
SASS PARTICULATE FILTER*
(μg)

U	<1	Dy	<1	Rh	<1	Cr	<5
Th	<1	Tb	<1	Ru	<1	V	<1
Bi	<1	Gd	<1	Mo	<2	Ti	<70
Pb	<600	Eu	<1	Nb	<1	Sc	<0.5
Tl	<1	Sm	<1	Zr	<6	Ca	<700
Hg		Nd	<1	Y	<0.5	K	<300
Au	<1	Pr	<1	Sr	<4	Cl	
Pt		Ce	2.6	Rb	<4	S	24,000
Ir	<1	La	<1	Br	<2	P	<50
Os	<1	Ba	<300	Se	4.9	Si	
Re	<1	Cs	<1	As	<6	Al	<2,000
W	<8	I	<1	Ge	<1	Mg	<100
Ta		Te	<1	Ga	<1	Na	12,000
Hf	<1	Sb	<3	Zn	860	F	<100
Lu	<1	Sn		Cu	210	B	<100
Yb	<1	In		Ni	180	Be	<1
Tm	<1	Cd	<8	Co	<1	Li	<4
Er	<1	Ag		Fe	400		
Ho	<1	Pd	<1	Mn	<20		

* Aqua regia digestion; no Parr bomb.

TABLE 25. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 301
XAD-2 RESIN
(μg)

U	<2	Dy	<3	Rh	<3	Cr	<40
Th	<2	Tb	<1	Ru	<2	V	<10
Bi	<2	Gd	<3	Mo	10	Ti	13
Pb	<200	Eu	<1	Nb	<3	Sc	<1
Tl	<3	Sm	<2	Zr	<6	Ca	1,200
Hg		Nd	<20	Y	<2	K	<30
Au	<2	Pr	<4	Sr	<20	Cl	<4,000
Pt		Ce	9.6	Rb	<20	S	72,000
Ir	<2	La	<4	Br	<5	P	65
Os	<2	Ba	<70	Se	<4	Si	
Re	<2	Cs	<2	As	<7	Al	880
W	<3	I	<2	Ge	<1	Mg	150
Ta		Te	<4	Ga	<1	Na	<6,000
Hf	<2	Sb	<7	Zn	580	F	<20
Lu	<1	Sn	<40	Cu	150	B	<2
Yb	<2	In		Ni	130	Be	<1
Tm	<1	Cd	29	Co	<4	Li	<4
Er	<2	Ag		Fe	<800		
Ho	<2	Pd	<2	Mn	<30		

TABLE 26. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 301
CONDENSATE/HNO_3 RINSE
(μg)

U	<5	Dy	<7	Rh	<2	Cr	1,100
Th	<5	Tb	<2	Ru	<2	V	<5
Bi	<5	Gd	<7	Mo	180	Ti	93
Pb	<200	Eu	<2	Nb	<5	Sc	<2
Tl	<5	Sm	<5	Zr	5	Ca	4.400
Hg		Nd	<5	Y	<2	K	1,400
Au	<2	Pr	<2	Sr	71	Cl	<1,000
Pt		Ce	15	Rb		S	12,000
Ir	<5	La	<5	Br	<2	P	1,100
Os	<5	Ba	64	Se	<7	Si	
Re	<5	Cs	<2	As	20	Al	220
W	<12	I	<2	Ge	<2	Mg	78
Ta		Te	<5	Ga	<2	Na	10,000
Hf	<5	Sb	100	Zn	<300	F	<70
Lu	<2	Sn		Cu	<800	B	<5
Yb	<5	In		Ni	<60	Be	<2
Tm	<2	Cd	<10	Co	7	Li	<2
Er	<5	Ag		Fe	2,600		
Ho	<2	Pd	<2	Mn	86		

TABLE 27. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 301
TOTAL EMISSIONS FOUND
(μg/m^3)

U	<0.08	Dy	<0.2	Rh	<0.07	Cr	13
Th	<0.08	Tb	<0.05	Ru	<0.06	V	<0.3
Bi	<0.08	Gd	<0.2	Mo	2.2	Ti	1-3
Pb	<12	Eu	<0.05	Nb	<0.1	Sc	<0.05
Tl	<0.1	Sm	<0.09	Zr	0.06-0.3	Ca	64
Hg		Nd	<0.4	Y	<0.06	K	16-30
Au	<0.06	Pr	<0.08	Sr	0.8-0.2	Cl	
Pt		Ce	0.31	Rb		S	12,000
Ir	<0.09	La	<0.2	Br	<0.1	P	14
Os	<0.09	Ba	0.6-5	Se	0.06-0.3	Si	
Re	<0.09	Cs	<0.06	As	0.23	Al	10-40
W	<0.4	I	<0.06	Ge	<0.05	Mg	2-4
Ta		Te	<0.2	Ga	<0.05	Na	320
Hf	<0.09	Sb	1.1	Zn	17	F	<3
Lu	<0.05	Sn		Cu	4.1-1,400	B	<1
Yb	<0.09	In		Ni	3.5	Be	<0.05
Tm	<0.05	Cd	0.3	Co	<0.3	Li	<0.2
Er	<0.09	Ag		Fe	34		
Ho	<0.06	Pd	<0.06	Mn	1.0		

TABLE 28. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 302
FUEL OIL
(ppm)

U	<0.01	Dy	<0.03	Rh	<0.1	Cr	<0.3
Th	<0.01	Tb	<0.05	Ru	<0.05	V	<0.04
Bi	<0.01	Gd	<0.05	Mo	<0.05	Ti	<0.1
Pb	<0.35	Eu	<0.05	Nb	<0.03	Sc	<0.02
Tl	<0.04	Sm	<0.05	Zr	<0.05	Ca	<4
Hg		Nd	<0.05	Y	<0.02	K	<2
Au	<0.02	Pr	<0.05	Sr	<0.06	Cl	<50
Pt		Ce	<0.05	Rb	<0.02	S	<100
Ir	<0.02	La	<0.05	Br	<0.05	P	0.2
Os	<0.02	Ba	<0.8	Se	<0.03	Si	
Re	<0.02	Cs	<0.04	As	<0.03	Al	<3
W	<0.03	I	0.04	Ge	<0.03	Mg	<0.4
Ta		Te	<0.5	Ga	<0.03	Na	<20
Hf	<0.02	Sb	<0.02	Zn	<2	F	<0.1
Lu	<0.01	Sn	<0.2	Cu	<3	B	<0.04
Yb	<0.02	In		Ni	3.8	Be	<0.01
Tm	2.01	Cd	<0.18	Co	<0.03	Li	<0.02
Er	<0.02	Ag		Fe	<5		
Ho	<0.01	Pd	<0.03	Mn	<0.3		

TABLE 29. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 302
SASS PARTICULATE FILTER*
(μg)

U	<1	Dy	<1	Rh	<1	Cr	15
Th	<1	Tb	<1	Ru	<1	V	<1
Bi	<1	Gd	<1	Mo	<2	Ti	<60
Pb	<400	Eu	<1	Nb	<1	Sc	<1
Tl	<1	Sm	<1	Zr	<10	Ca	<800
Hg		Nd	<1	Y	<1	K	<300
Au	<1	Pr	<1	Sr	<5	Cl	
Pt		Ce	<2	Rb	<4	S	34,000
Ir	<1	La	<1	Br	<2	P	<50
Os	<1	Ba	<30	Se	<3	Si	
Re	<1	Cs	<1	As	<5	Al	<800
W	<1	I	<1	Ge	<1	Mg	<100
Ta		Te	<1	Ga	<1	Na	<6,000
Hf	<1	Sb	<1	Zn	<400	F	<30
Lu	<1	Sn	<10	Cu	350	B	<10
Yb	<1	In		Ni	<350	Be	<0.5
Tm	<1	Cd	<9	Co	<1	Li	<5
Er	<1	Ag		Fe	750		
Ho	<1	Pd	<1	Mn	<15		

*Aqua regia digestion; no Parr bomb.

TABLE 30. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 302
XAD-2 RESIN
(μg)

U	<3	Dy	<10	Rh	<2	Cr	<40
Th	<3	Tb	<3	Ru	<2	V	<20
Bi	<3	Gd	<10	Mo	8.5	Ti	26
Pb	<200	Eu	<3	Nb	<3	Sc	<7
Tl	<6	Sm	<6	Zr	<2	Ca	21,000
Hg		Nd	<10	Y	<2	K	270
Au	<3	Pr	23	Sr	27	Cl	<12,000
Pt		Ce	<6	Rb	<30	S	30,000
Ir	<6	La	<3	Br	<8	P	250
Os	<6	Ba	77	Se	<6	Si	
Re	<6	Cs	<3	As	<50	Al	<13
W	<5	I	<3	Ge	<6	Mg	<500
Ta		Te	<10	Ga	<6	Na	7,500
Hf	<6	Sb	<7	Zn	1,300	F	<40
Lu	<3	Sn	<70	Cu	350	B	<4
Yb	<3	In		Ni	250	Be	<3
Tm	<3	Cd	370	Co	<7	Li	<4
Er	<6	Ag		Fe	<1,300		
Ho	<3	Pd	<3	Mn	<50		

TABLE 31. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 302
TOTAL EMISSIONS FOUND
(μg/m³)

U	<0.04
Th	<0.04
Bi	<0.04
Pb	<6
Tl	<0.07
Hg	
Au	<0.04
Pt	
Ir	<0.07
Os	<0.07
Re	<0.07
W	<0.06
Ta	
Hf	<0.07
Lu	<0.04
Yb	<0.04
Tm	<0.04
Er	<0.07
Ho	<0.04
Dy	<0.2
Tb	<0.04
Gd	<0.2
Eu	<0.04
Sm	<0.07
Nd	<0.2
Pr	<0.04
Ce	<0.08
La	<0.04
Ba	0.7-1.1
Cs	<0.04
I	<0.04
Te	<0.2
Sb	<0.08
Sn	<0.8
In	
Cd	3.7
Ag	
Pd	<0.04
Rh	<0.03
Ru	<0.03
Mo	0.085
Nb	<0.04
Zr	<0.2
Y	<0.03
Sr	0.30
Rb	<0.4
Br	<0.1
Se	<0.1
As	<0.7
Ge	<0.07
Ga	<0.07
Zn	12-17
Cu	7.0
Ni	2.5-6.0
Co	<0.08
Fe	7.5-22
Mn	<0.7
Cr	0.15-0.6
V	<0.3
Ti	0.2-0.9
Sc	<0.08
Ca	210
K	2.7-6.0
Cl	
S	640
P	2.5
Si	
Al	<9
Mg	<6
Na	75-140
F	<0.7
B	<0.15
Be	<0.04
Li	<0.09

TABLE 32. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 303
FUEL OIL
(ppm)

U	<0.02
Th	<0.02
Bi	<0.02
Pb	0.38
Tl	<0.02
Hg	
Au	<0.02
Pt	
Ir	<0.02
Os	<0.03
Re	<0.02
W	<0.04
Ta	
Hf	<0.03
Lu	<0.02
Yb	<0.04
Tm	<0.02
Er	<0.03
Ho	<0.02
Dy	<0.04
Tb	<0.01
Gd	<0.04
Eu	<0.02
Sm	<0.03
Nd	0.043
Pr	<0.0067
Ce	<0.05
La	<0.02
Ba	<0.8
Cs	<0.01
I	<0.1
Te	<0.01
Sb	<0.03
Sn	
In	
Cd	0.052
Ag	
Pd	<0.02
Rh	<0.04
Ru	<0.02
Mo	<0.05
Nb	<0.03
Zr	<0.05
Y	<0.1
Sr	<0.04
Rb	<0.11
Br	<0.02
Se	<0.067
As	<0.03
Ge	<0.1
Ga	<0.01
Zn	<1.4
Cu	<2
Ni	<4
Co	<0.01
Fe	<5
Mn	<0.2
Cr	<0.3
V	<0.2
Ti	0.34
Sc	<0.01
Ca	<3
K	<1
Cl	<20
S	170
P	0.22
Si	
Al	<7
Mg	0.52
Na	<15
F	<0.01
B	<0.01
Be	<0.01
Li	<0.007

TABLE 33. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 303
SASS PARTICULATE FILTER*
(μg)

U	<0.3	Dy	<0.7	Rh	<0.7	Cr	27
Th	<0.3	Tb	<0.2	Ru	<0.3	V	0.76
Bi	<1	Gd	<0.7	Mo	2.2	Ti	81
Pb	1,200	Eu	<0.3	Nb	<0.1	Sc	<0.2
Tl	<0.3	Sm	<0.5	Zr	<15	Ca	<700
Hg		Nd	1.1	Y	<0.4	K	<300
Au	<0.3	Pr	<0.2	Sr	5.4	Cl	
Pt		Ce	<0.6	Rb	<5	S	36,000
Ir	<0.3	La	<0.4	Br	<1	P	<150
Os	<0.5	Ba	<30	Se	3.4	Si	
Re	<0.3	Cs	<0.3	As	<3	Al	<900
W	<0.7	I	<0.2	Ge	<0.2	Mg	<150
Ta		Te	<0.3	Ga	<0.2	Na	<2,500
Hf	<0.5	Sb	<3	Zn	3,000	F	<0.2
Lu	<0.3	Sn	12	Cu	940	B	<40
Yb	<0.7	In		Ni	<500	Be	<0.03
Tm	<0.3	Cd	<12	Co	<0.9	Li	<1.5
Er	<0.5	Ag		Fe	<1,000		
Ho	<0.3	Pd	<0.3	Mn	47		

*Aqua regia digestion; no Parr bomb.

TABLE 34. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 303
TOTAL EMISSIONS FOUND
($\mu g/m^3$)

U	<0.004	Dy	<0.009	Rh	<0.009	Cr	0.34
Th	<0.004	Tb	<0.003	Ru	<0.004	V	0.0095
Bi	<0.1	Gd	<0.009	Mo	0.028	Ti	1.0
Pb	15	Eu	<0.004	Nb	<0.002	Sc	<0.003
Tl	<0.004	Sm	<0.006	Zr	<0.2	Ca	<9
Hg		Nd	0.014	Y	<0.005	K	<4
Au	<0.004	Pr	<0.003	Sr	0.068	Cl	
Pt		Ce	<0.008	Rb	<0.06	S	450
Ir	<0.004	La	<0.005	Br	<0.02	P	<2
Os	<0.006	Ba	<0.4	Se	0.043	Si	
Re	<0.004	Cs	<0.004	As	<0.04	Al	<12
W	<0.009	I	<0.003	Ge	<0.003	Mg	<2
Ta		Te	<0.004	Ga	<0.003	Na	<30
Hf	<0.006	Sb	<0.04	Zn	38	F	<0.003
Lu	<0.004	Sn	0.14	Cu	12	B	<0.5
Yb	<0.009	In		Ni	<7	Be	<0.004
Tm	<0.004	Cd	<0.15	Co	<0.01	Li	<0.02
Er	<0.006	Ag		Fe	<13		
Ho	<0.004	Pd	<0.004	Mn	0.59		

TABLE 35. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 304
FUEL OIL
(ppm)

U	<0.02	Dy	<0.04	Rh	<0.04	Cr	<0.4
Th	<0.02	Tb	<0.01	Ru	<0.02	V	0.059
Bi	<0.02	Gd	<0.04	Mo	0.10	Ti	<0.2
Pb	1.3	Eu	<0.02	Nb	<0.03	Sc	<0.01
Tl	<0.02	Sm	<0.03	Zr	<0.05	Ca	<8
Hg		Nd	0.061	Y	<0.01	K	<1.4
Au	<0.02	Pr	0.016	Sr	<0.08	Cl	<10
Pt		Ce	0.11	Rb	<0.04	S	290
Ir	<0.02	La	0.025	Br	<0.04	P	0.21
Os	<0.03	Ba	0.81	Se	0.066	Si	
Re	<0.02	Cs	<0.01	As	0.055	Al	27
W	<0.04	I	<0.01	Ge	<0.01	Mg	0.27
Ta		Te	<0.02	Ga	<0.03	Na	<30
Hf	<0.03	Sb	0.55	Zn	1.4	F	<0.1
Lu	<0.02	Sn	<2	Cu	3.3	B	<0.023
Yb	<0.02	In		Ni	<4.0	Be	<0.012
Tm	<0.02	Cd	0.50	Co	0.027	Li	<0.01
Er	<0.03	Ag		Fe	54		
Ho	<0.02	Pd	<0.02	Mn	0.47		

TABLE 36. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 304
SASS PARTICULATE FILTER*
(μg)

U	<0.3	Dy	<0.7	Rh	<0.7	Cr	<8
Th	<0.3	Tb	<0.2	Ru	<0.3	V	1.2
Bi	<0.3	Gd	<0.7	Mo	3.7	Ti	<60
Pb	<120	Eu	<0.3	Nb	<0.2	Sc	<0.2
Tl	<0.3	Sm	<0.5	Zr	<10	Ca	<17
Hg		Nd	<1	Y	<0.2	K	<200
Au	<0.3	Pr	<0.2	Sr	<3	Cl	
Pt		Ce	<0.7	Rb	<5	S	11,000
Ir	<0.3	La	<0.2	Br	<1	P	<20
Os	<0.5	Ba	<30	Se	<0.3	Si	
Re	<0.3	Cs	4.0	As	<3	Al	<1,300
W	<0.7	I	<0.2	Ge	<0.2	Mg	<50
Ta		Te	<0.3	Ga	<0.2	Na	5,000
Hf	<0.5	Sb	<0.5	Zn	350	F	<3
Lu	<0.3	Sn	<15	Cu	<500	B	110
Yb	<0.7	In		Ni	<320	Be	<0.2
Tm	<0.3	Cd	<0.3	Co	2.2	Li	<1
Er	<0.5	Ag		Fe	6,400		
Ho	<0.3	Pd	<0.3	Mn	18		

*Aqua regia digestion; no Parr bomb.

TABLE 37. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 304
XAD-2 RESIN
(μg)

U	<3	Dy	<6	Rh	<6	Cr	<10
Th	<3	Tb	<2	Ru	<3	V	<1
Bi	<3	Gd	<6	Mo	5.5	Ti	<2
Pb	<20	Eu	<3	Nb	8.1	Sc	<2
Tl	<3	Sm	<4	Zr	7.9	Ca	<7,000
Hg		Nd	<8	Y	<2	K	<100
Au	<3	Pr	<1	Sr	<10	Cl	<4,000
Pt		Ce	<5	Rb	<4	S	<16,000
Ir	<3	La	9.5	Br	<3	P	<86
Os	<4	Ba	<50	Se	<2	Si	
Re	<3	Cs	<1	As	<2	Al	<300
W	<6	I	<2	Ge	<2	Mg	<300
Ta		Te	<3	Ga	2.5	Na	<2,500
Hf	<4	Sb	<5	Zn	<120	F	<0.2
Lu	<3	Sn	<16	Cu	<160	B	<0.2
Yb	<6	In		Ni	<160	Be	<0.03
Tm	<3	Cd	<7	Co	<1	Li	<0.9
Er	<4	Ag		Fe	<200		
Ho	<3	Pd	<3	Mn	<6		

TABLE 38. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED FURNACE: SITE 304
TOTAL EMISSIONS FOUND
(μg)

U	<0.04	Dy	<0.08	Rh	<0.08	Cr	<0.3
Th	<0.04	Tb	<0.03	Ru	<0.04	V	0.016-0.026
Bi	<0.04	Gd	<0.08	Mo	0.12	Ti	<0.8
Pb	<2	Eu	<0.04	Nb	0.11	Sc	<0.03
Tl	<0.04	Sm	<0.05	Zr	0.10-0.20	Ca	<90
Hg		Nd	<0.1	Y	<0.03	K	<4
Au	<0.04	Pr	<0.01	Sr	<0.2	Cl	
Pt		Ce	0.07	Rb	<0.1	S	140-390
Ir	<0.04	La	0.12	Br	<0.05	P	<1.5
Os	<0.05	Ba	<1	Se	<0.03	Si	
Re	<0.04	Cs	0.052	As	<0.07	Al	<20
W	<0.08	I	<0.03	Ge	<0.03	Mg	<5
Ta		Te	<0.04	Ga	0.032	Na	65-90
Hf	<0.05	Sb	<0.07	Zn	4.5-5.8	F	<0.04
Lu	<0.04	Sn	<0.4	Cu	<9	B	1.4
Yb	<0.08	In		Ni	<8	Be	<0.003
Tm	<0.04	Cd	<0.09	Co	0.029-0.038	Li	<0.03
Er	<0.05	Ag		Fe	83		
Ho	<0.04	Pd	<0.04	Mn	0.23		

TABLE 39. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS GAS-FIRED FURNACE ($\mu g/m^3$)

Sample	Hg	Sb	As
XAD-2 resin			
SITE 100	0.03	<0.5	<0.11
SITE 101	0.25	<0.5	<0.13
SITE 102	3.14	<0.5	<0.12
SITE 103	4.02	<0.5	<0.11
SITE 104	1.04	<0.5	<0.12
Composite*			
SITE 100	ND†	<0.17	<0.04
SITE 101	ND	<0.3	0.36
SITE 102	ND	3.8	<0.04
SITE 103	ND	<0.11	<0.03
SITE 104	ND	<0.11	<0.03
APS impinger			
SITE 100	ND	<0.05	<0.03
SITE 101	ND	<0.12	<0.03
SITE 102	ND	<0.06	<0.03
SITE 103	ND	<0.04	<0.02
SITE 104	ND	<0.06	<0.03

*Composite of condensate, HNO_3 wash of XAD-2 module and H_2O_2 impinger.

†ND--Not detected indicates sample less than blank concentration.

TABLE 40. GAS CHROMATOGRAPHY* FOR INORGANIC GASES

Sample	CO ppm	O_2 %	CO_2 %
Gas-fired furnace†			
SITE 100	<500	16.7	6.4
SITE 101	<500	12.9	1.4
SITE 102	<500	19.5	3.0
SITE 103	<500	19.1	1.7
SITE 104	<500	16.8	1.1
Oil-fired furnace‡			
SITE 300	<0.2	17.2	3.8
SITE 301	<0.2	17.4	3.7
SITE 302	<0.2	19.6	1.2
SITE 303	<0.2	17.3	2.9
SITE 304	<0.2	17.5	2.6

*Separation by GC with thermal conductivity detector.

†Collection by Tedlar bag.

‡Collection by evacuated flask.

TABLE 41. CHEMILUMINESCENCE FOR NO_x* GAS-FIRED FURNACE FLUE GAS

Sample	NO_x concentrations	
	mg/m^3	$lbs/10^6$ Btu
SITE 100	25.3	0.054
SITE 101	9.4	0.011
SITE 102	18.8	0.027
SITE 103	12.5	0.026
SITE 104	9.3	0.011

*Theta sensor used for determination electrochemically. Collection by Tedlar bags.

TABLE 42. GAS CHROMATOGRAPHY FOR C_1-C_6/C_7*
GAS-FIRED FURNACE

Sample		Range	Weight µg/m^3	No. of peaks observed
SITE 100	GC1	160-100	0	
	GC2	100-50	0	
	GC3	50-0	0	
	GC4	0-30	0	
	GC5	30-60	0	
	GC6	60-90	0	
	GC7	90-100	Not reported	
SITE 101	GC1	160-100	0	
	GC2	100-50	0	
	GC3	50-0	0	
	GC4	0-30	0	
	GC5	30-60	0	
	GC6	60-90	0	
	GC7	90-100	Not reported	
SITE 102	GC1	160-100	0	
	GC2	100-50	0	
	GC3	50-0	0	
	GC4	0-30	0	
	GC5	30-60	0	
	GC6	60-90	0	
	GC7	90-100	Not reported	
SITE 103†	GC1	160-100	39,400	
	GC2	100-50	37,800	
	GC3	50-0	55,500	
	GC4	0-30	0	
	GC5	30-60	0	
	GC6	60-90	0	
	GC7	90-100	Not reported	
SITE 104	GC1	160-100	0	
	GC2	100-50	0	
	GC3	50-0	0	
	GC4	0-30	0	
	GC5	30-60	0	
	GC6	60-90	0	
	GC7	90-100	Not reported	

*Flame ionization detection; detection limits 1.0 ppm (1,000 µg/m^3).

†Furnace appears to have malfunctioned resulting in excess emissions of C_1, C_2, and C_3.

TABLE 43. GAS CHROMATOGRAPHY FOR C_7-C_{17} GAS-FIRED FURNACE--COMBINED EXTRACTS*

Sample	Range	Volatile weight, μg/m³	No. of peaks	Gravimetric nonvolatile weight, μg/m³	Total organic, μg/m³
SITE 100	GC7 90-110	Not reported			
	GC8 110-140	<10			
	GC9 140-160	<10			
	GC10 160-180	1,490			
	GC11 180-200	560			
	GC12 200-220	60			
	>GC17			400	
	GC1 - >GC17				~2,500
SITE 101	GC7 90-110	Not reported			
	GC8 110-140	<10			
	GC9 140-160	<10			
	GC10 160-180	<10			
	GC11 180-200	<10			
	GC12 200-220	<10			
	>GC17			1,240	
	GC1 - >GC17				~1,240
SITE 102	GC7 90-110	Not reported			
	GC8 110-140	---			
	GC9 140-160	---			
	GC10 160-180	---			
	GC11 180-200	---			
	GC12 200-220				
	>GC17			780	
	GC1 - >GC17				>780
SITE 103	GC7 90-110	Not reported			
	GC8 110-140	<10			
	GC9 140-160	<10			
	GC10 160-180	770			
	GC11 180-200	680			
	GC12 200-220	<10			
	>GC17			480	
	GC1 - >GC17				~1,900
SITE 104	GC7 90-110	Not reported			
	GC8 110-140	<10			
	GC9 140-160	<10			
	GC10 160-180	1,960			
	GC11 180-200	2,680			
	GC12 200-220	370			
	>GC17			920	
	GC1 - >GC17				~5,900

See footnote at end of table.

TABLE 43 (con.)

Sample	Range	Volatile weight, $\mu g/m^3$	No. of peaks	Gravimetric nonvolatile weight, $\mu g/m^3$	Total organic, $\mu g/m^3$
Average Gas-fired sites 100-104	GC7 90-110	Not reported			
	GC8 110-140	<10			
	GC9 140-160	<10			
	GC10 160-180	1,060			
	GC11 180-200	980			
	GC12 200-220	110			
	>GC17			760	
	GC1 - >GC17				~2,500

*CH_2Cl_2 extract of XAD-2 resin, rinses, etc.

TABLE 44. GAS CHROMATOGRAPHY FOR C_7-C_{17} GAS-FIRED FURNACE--SASS TRAIN SAMPLES

Sample	Range	Volatile weight, mg/m^3	No. of peaks	Gravimetric nonvolatile weight, mg/m^3	Total organic weight
Probe solvent rinse (PR-0)					
SITE 100	C_7-C_{12}	<0.010			
	$>C_{17}$			0	
SITE 101	C_7-C_{12}	<0.010			
	$>C_{17}$			0.14	
SITE 102	C_7-C_{12}	---			
	$>C_{17}$			0.025	
SITE 103	C_7-C_{12}	0.36			
	$>C_{17}$			0.03	
SITE 104	C_7-C_{12}	<0.010			
	$>C_{17}$			0	
Solvent XAD-2 module rinse (MR-0)					
SITE 100	C_7-C_{12}	<0.010			
	$>C_{17}$			0.18	
SITE 101	C_7-C_{12}	<0.010			
	$>C_{17}$			0.81	
SITE 102	C_7-C_{12}	---			
	$>C_{17}$			0.47	
SITE 103	C_7-C_{12}	<0.010			
	$>C_{17}$			0.27	
SITE 104	C_7-C_{12}	<0.010			
	$>C_{17}$			0.86	
XAD-2 resin--solvent extract (XR-S)					
SITE 100	C_7-C_{12}	2.11			
	$>C_{17}$			0.22	
SITE 101	C_7-C_{12}	<0.010			
	$>C_{17}$			0.29	
SITE 102	C_7-C_{12}	---			
	$>C_{17}$			0.28	
SITE 103	C_7-C_{12}	1.09			
	$>C_{17}$			0.18	
SITE 104	C_7-C_{12}	5.01			
	$>C_{17}$			0.06	

TABLE 45. GAS CHROMATOGRAPHY FOR C_1-C_6/C_7* OIL-FIRED FURNACE

Sample		Range	Weight µg/m³	No. of peaks observed
SITE 300	GC1	160-100	9,600	
	GC2	100-50	1,700	
	GC3	50-0	<200	
	GC4	0-30	<300	
	GC5	30-60	<300	
	GC6	60-90	<400	
	GC7	90-100	Not reported	
SITE 301	GC1	160-100	2,700	
	GC2	100-50	1,900	
	GC3	50-0	4,400	
	GC4	0-30	<300	
	GC5	30-60	<300	
	GC6	60-90	<400	
	GC7	90-100	Not reported	
SITE 302	GC1	160-100	2,000	
	GC2	100-50	400	
	GC3	50-0	<200	
	GC4	0-30	<300	
	GC5	30-60	<300	
	GC6	60-90	<400	
	GC7	90-100	Not reported	
SITE 303	GC1	160-100	1,800	
	GC2	100-50	<100	
	GC3	50-0	<200	
	GC4	0-30	<300	
	GC5	30-60	<300	
	GC6	60-90	<400	
	GC7	90-100	Not reported	
SITE 304	GC1	160-100	2,800	
	GC2	100-50	140	
	GC3	50-0	<200	
	GC4	0-30	<300	
	GC5	30-60	<300	
	GC6	60-90	<400	
	GC7	90-100	Not reported	
Average Oil-fired SITE 300-304	GC1	160-100	3,800	
	GC2	100-50	850	
	GC3	50-0	>1,000	
	GC4	0-30	<300	
	GC5	30-60	<300	
	GC6	60-90	<400	
	GC7	90-100	Not reported	

*Document states analysis by GC with FID. Lower detection limits must have been realized with oil-fired samples as opposed to 1,000 µg/m³ for gas-fired samples. (See Table 42.)

TABLE 46. GAS CHROMATOGRAPHY FOR C_7-C_{17} OIL-FIRED FURNACE--COMBINED EXTRACTS*

Sample	Range	Volatile weight, μg/m³	No. of peaks	Gravimetric nonvolatile weight, μg/m³	Total organic, μg/m³
SITE 300	GC7 90-110	Not reported			
	GC8 110-140	6.3			
	GC9 140-160	4.9			
	GC10 160-180	46			
	GC11 180-200	25			
	GC12 200-220	1.3			
	GC12 - GC17	86			
	>GC17			650	
	GC1 - >GC17				12,000-13,000
SITE 301	GC7 90-110	Not reported			
	GC8 110-140	1.2			
	GC9 140-160	3.0			
	GC10 160-180	14			
	GC11 180-200	6.6			
	GC12 200-220	33			
	GC12 - GC17	122			
	>GC17			1,800	
	GC1 - >GC17				11,000-12,000
SITE 302	GC7 90-110	Not reported			
	GC8 110-140	7.5			
	GC9 140-160	9.0			
	GC10 160-180	26			
	GC11 180-200	44			
	GC12 200-220	45			
	GC12 - GC17	429			
	>GC17			290-560	
	GC1 - >GC17				3,000-4,700
SITE 303	GC7 90-110	Not reported			
	GC8 110-140	1.2			
	GC9 140-160	15			
	GC10 160-180	43			
	GC11 180-200	47			
	GC12 200-220	65			
	GC12 - GC17	384			
	>GC17			1,200-1,400	
	GC1 - >GC17				3,600-5,100

See footnote at end of table.

TABLE 46 (con.)

Sample	Range	Volatile weight, μg/m^3	No. of peaks	Gravimetric nonvolatile weight, μg/m^3	Total organic, μg/m^3
SITE 304	GC7 90-110	Not reported			
	GC8 110-140	5.2			
	GC9 140-160	7.3			
	GC10 160-180	43			
	GC11 180-200	49			
	GC12 200-220	81			
	GC12 - GC17	134			
	>GC17			6,000	
	GC1 - >GC17				9,300-10,500
Average Oil-fired sites 300-304	GC7 90-110	Not reported			
	GC8 110-140	4.3			
	GC9 140-160	7.8			
	GC10 160-180	34			
	GC11 180-200	34			
	GC12 200-220	45			
	GC12 - GC17	360			
	>GC17			2,000-2,400	
	GC1 - >GC17				7,800-9,100

*CH_2Cl_2 extract of XAD-2 resin, rinses, etc.

TABLE 47. GAS CHROMATOGRAPHY FOR C_7-C_{17}* OIL-FIRED FURNACE--SASS TRAIN SAMPLES

Sample type	Site	Volatile C_7-C_{12}		Nonvolatile $>C_{17}$		Volume sampled, m^3	Total organics mg/m^3
		Total µg	mg/m^3	Total µg	mg/m^3		
Probe solvent rinse (PR-0)	300	<31		<1,000		91.9	---
	301	<217		<5,000		88.1	---
	302	<140		<13,000		104	---
	303	<116		<6,000		80.6	---
	304	<285		114,000	1.49	76.7	1.49
Solvent XAD-2 module rinse (MR-0)	300	<31		<1,000		91.9	---
	301	644	0.0073	20,200	0.229	88.1	0.236
	302	<140		<13,000		104	---
	303	<116		<6,000		80.6	---
	304	<285		14,900	0.194	76.7	0.194
Condensate--solvent extract (CD-S)	300	<300		15,600	0.170	91.9	0.170
	301	<34		87,800	0.997	88.1	0.997
	302	1,880	0.0213	<2,000		104	0.021
	303	1,840	0.0228	<6,000		80.6	0.023
	304	<341		92,700	1.21	76.7	1.21
XAD-2 resin--solvent extract (XR-S)	300	15,500	0.167	43,000	0.469	91.9	0.636
	301	15,400	0.175	52,600	0.597	88.1	0.772
	302	56,600	0.544	30,200	0.290	104	0.834
	303	42,900	0.532	96,600	1.20	80.6	1.732
	304	24,600	0.321	237,000	3.09	76.7	3.411

*All values have been blank-corrected. (Note that this is incorrect data format. Data was not broken down.)

†All numbers reported as "less than" (<) are sample values found to be less than the blank value; number reported is value of blank.

TABLE 48. GAS CHROMATOGRAPHY FOR C_7-C_{12}*
OIL-FIRED FURNACE--SASS TRAIN SAMPLES, SITE 300

Sample	Range	Volatile weight, $\mu g/m^3$	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Probe solvent rinse (PR-0)	GC7 90-110	Not reported			
	GC8 110-140	0			
	GC9 140-160	†			
	GC10 160-180	†			
	GC11 180-200	0			
	GC12 200-220	0			
XAD-2 module rinse (MR-0)	GC7 90-110	Not reported			
	GC8 110-140	0			
	GC9 140-160	0.422			
	GC10 160-180	0.984			
	GC11 180-200	1.14			
	GC12 200-220	0			
Condensate extract (CD-S)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	†			
	GC10 160-180	†			
	GC11 180-200	†			
	GC12 200-220	†			
XAD-2 resin extract (XR-S)	GC7 90-110	Not reported			
	GC8 110-140	6.33			
	GC9 140-160	4.33			
	GC10 160-180	45.4			
	GC11 180-200	23.8			
	GC12 200-220	1.31			

*All values have been blank-corrected.

†Less than blank.

TABLE 49. GAS CHROMATOGRAPHY FOR C_7-C_{12}* OIL-FIRED FURNACE--SASS TRAIN SAMPLES, SITE 301

Sample	Range		Volatile weight, $\mu g/m^3$	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Probe solvent rinse (PR-0)	GC7	90-110	Not reported			
	GC8	110-140	†			
	GC9	140-160	†			
	GC10	160-180	†			
	GC11	180-200	0			
	GC12	200-220	0			
XAD-2 module rinse (MR-0)	GC7	90-110	Not reported			
	GC8	110-140	0.0276			
	GC9	140-160	†			
	GC10	160-180	0.221			
	GC11	180-200	0.286			
	GC12	200-220	0.110			
Condensate extract (CD-S)	GC7	90-110	Not reported			
	GC8	110-140	4.96			
	GC9	140-160	0.110			
	GC10	160-180	†			
	GC11	180-200	†			
	GC12	200-220	†			
XAD-2 resin extract (XR-S)	GC7	90-110	Not reported			
	GC8	110-140	6.95			
	GC9	140-160	2.75			
	GC10	160-180	13.7			
	GC11	180-200	6.36			
	GC12	200-220	32.6			

*All values have been blank-corrected.

†Less than blank.

TABLE 50. GAS CHROMATOGRAPHY FOR C_7-C_{12}*
OIL-FIRED FURNACE--SASS TRAIN SAMPLES, SITE 302

Sample	Range	Volatile weight, $\mu g/m^3$	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Probe solvent rinse (PR-0)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	†			
	GC10 160-180	0.0167			
	GC11 180-200	0			
	GC12 200-220	0			
XAD-2 module rinse (MR-0)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	†			
	GC10 160-180	0.234			
	GC11 180-200	0.395			
	GC12 200-220	0.420			
Condensate extract (CD-S)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	0.396			
	GC10 160-180	0.981			
	GC11 180-200	0			
	GC12 200-220	13.5			
XAD-2 resin extract (XR-S)	GC7 90-110	Not reported			
	GC8 110-140	7.51			
	GC9 140-160	8.65			
	GC10 160-180	24.7			
	GC11 180-200	43.4			
	GC12 200-220	31.0			

*All values have been blank-corrected.

†Less than blank.

TABLE 51. GAS CHROMATOGRAPHY FOR C_7-C_{12}* OIL-FIRED FURNACE--SASS TRAIN SAMPLES, SITE 303

Sample	Range	Volatile weight, μg/m^3	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Probe solvent rinse (PR-0)	GC7 90-110	Not reported			
	GC8 110-140	0			
	GC9 140-160	†			
	GC10 160-180	0.150			
	GC11 180-200	0			
	GC12 200-220	†			
XAD-2 module rinse (MR-0)	GC7 90-110	Not reported			
	GC8 110-140	0			
	GC9 140-160	†			
	GC10 160-180	0.242			
	GC11 180-200	0.023			
	GC12 200-220	†			
Condensate extract (CD-S)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	†			
	GC10 160-180	†			
	GC11 180-200	†			
	GC12 200-220	†			
XAD-2 resin extract (XR-S)	GC7 90-110	Not reported			
	GC8 110-140	11.6			
	GC9 140-160	14.6			
	GC10 160-180	41.6			
	GC11 180-200	46.5			
	GC12 200-220	64.7			

*All values have been blank-corrected.

†Less than blank.

TABLE 52. GAS CHROMATOGRAPHY FOR C_7-C_{12}*
OIL-FIRED FURNACE--SASS TRAIN SAMPLES, SITE 304

Sample	Range	Volatile weight, μg/m^3	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Probe solvent rinse (PR-0)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	†			
	GC10 160-180	†			
	GC11 180-200	0			
	GC12 200-220	0			
XAD-2 module rinse (MR-0)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	†			
	GC10 160-180	0.567			
	GC11 180-200	0.231			
	GC12 200-220	0.122			
Condensate extract (CD-S)	GC7 90-110	Not reported			
	GC8 110-140	†			
	GC9 140-160	†			
	GC10 160-180	0.131			
	GC11 180-200	0.335			
	GC12 200-220	†			
XAD-2 resin extract (XR-S)	GC7 90-110	Not reported			
	GC8 110-140	5.23			
	GC9 140-160	7.00			
	GC10 160-180	39.3			
	GC11 180-200	48.9			
	GC12 200-220	75.0			

*All values have been blank-corrected.

†Less than blank.

TABLE 53. LC FRACTIONATION--GAS-FIRED FURNACE CH_2Cl_2 EXTRACT OF XAD-2 RESIN (NONVOLATILE)

	SITE 101				SITE 102			
Fraction	TCO	Grav µg/m³	Total	Total	TCO	Grav µg/m³	Total	Total
LC 1		150				110		
LC 2		66				<0.4		
LC 3		23				38		
LC 4		18				23		
LC 5		2				76		
LC 6		87				110		
LC 7		44				<0.4		
LC 8		---				---		

	SITE 103			
Fraction	TCO	Grav µg/m³	Total	Total
LC 1		<0.8		
LC 2		100		
LC 3		61		
LC 4		520		
LC 5		<0.8		
LC 6		<0.8		
LC 7		150		
LC 8		---		

TABLE 54. LC FRACTIONATION--GAS-FIRED FURNACE CONDENSATE EXTRACT AND MODULE RINSE (NONVOLATILE)

	SITE 101				SITE 102			
Fraction	TCO	Grav $\mu g/m^3$	Total	Total	TCO	Grav $\mu g/m^3$	Total	Total
LC 1		<0.8				<0.5		
LC 2		<0.8				<0.5		
LC 3		12				<0.5		
LC 4		430				280		
LC 5		110				100		
LC 6		120				72		
LC 7		140				16		
LC 8		---				---		

	SITE 103			
Fraction	TCO	Grav $\mu g/m^3$	Total	Total
LC 1		N.R.		
LC 2		N.R.		
LC 3		N.R.		
LC 4		N.R.		
LC 5		N.R.		
LC 6		N.R.		
LC 7		N.R.		
LC 8		N.R.		

N.R. = not reported.

TABLE 55. LC FRACTIONATION--OIL-FIRED FURNACE CH_2CL_2 EXTRACT OF XAD-2 RESIN (NONVOLATILE)

	SITE 300				SITE 301			
Fraction	TCO $\mu g/m^3$	Grav $\mu g/m^3$	Total $\mu g/m^3$	Total	TCO $\mu g/m^3$	Grav $\mu g/m^3$	Total $\mu g/m^3$	Total
LC 1	43	88	131		68	30	98	
LC 2	36	<0.5	36		16	23	39	
LC 3	17	<0.5	17		15	35	50	
LC 4	15	24	39		20	15	35	
LC 5	17	107	124		36	23	59	
LC 6	45	91	126		1.4	402	416	
LC 7	0.2	157	157		9	65	74	
LC 8	---	---	---		<0.2	<0.6	<0.8	

	SITE 302				SITE 303			
Fraction	TCO $\mu g/m^3$	Grav $\mu g/m^3$	Total $\mu g/m^3$	Total	TCO $\mu g/m^3$*	Grav $\mu g/m^3$†	Total $\mu g/m^3$	Total
LC 1	91	1	92		<0.5	266	266	
LC 2	62	2	64		10	40	50	
LC 3	48	5.5	54		84	288	312	
LC 4	84	4.4	88		135	152	287	
LC 5	179	10	189		155	152	307	
LC 6	101	17	118		144	342	486	
LC 7	<0.6	250	250		29	19	48	
LC 8	<0.6	<0.3	<0.9		<0.5	<1	<1.5	

See footnotes at end of table.

TABLE 55 (con.)

	SITE 304			
Fraction	TCO µg/m^3†	Grav µg/m^3‡	Total µg/m^3	Total
LC 1	250	2,250	2,500	
LC 2	27	140	170	
LC 3	23	280	300	
LC 4	19	400	420	
LC 5	5	280	285	
LC 6	4	730	730	
LC 7	<0.3	400	400	
LC 8	<0.3	<10	<10	

*Four percent of the volatiles were from the condensate extract, remainder were from the XAD-2 resin.

†XAD-2 resin.

‡Four percent of the nonvolatiles were from the solvent module rinse, 27 percent from the condensate and the remainder from the XAD-2 resin.

TABLE 56. IR REPORT
SAMPLE: GAS-FIRED FURNACE

Quote from text:

"IR analysis was found to be impossible due to the insufficient amounts of material available for the taking of clean, interpretable spectra."

TABLE 57. IR REPORT*
SAMPLE: OIL-FIRED FURNACE

LC	SITE 300	SITE 301	SITE 302†
LC 1	Alkanes	Aliphatics	---
LC 2	Unknown	Aliphatics and halogen, substituted aliphatics	---
LC 3	Unknown	Substituted aliphatics	---
LC 4	R-CN, substituted aliphatics	Substituted aliphatics	---
LC 5	Substituted aliphatics and ethers	Substituted aliphatics	---
LC 6	Substituted aliphatics and alcohols	Aliphatics and/or aromatic esters and ketones and phenols	---
LC 7	Substituted aliphatics and carboxylic acids	Aliphatic acids	---
LC 8	Same as LC 7	Substituted aliphatic acids	---

See footnotes at end of table.

TABLE 57 (con.)

LC	SITE 303	SITE 304
LC 1	Aliphatics	Aliphatics and aromatics
LC 2	Substituted aliphatics	Aliphatics and aromatics
LC 3	Substituted aliphatics, ethers, esters, and ketones	Substituted aliphatics, ethers esters, and ketones
LC 4	Same as LC 3	Same as LC 3
LC 5	Ethers, esters, and ketone	Carbonyl compounds and aromatics
LC 6	Ethers, esters, and ketones	Same as LC 5
LC 7	Aliphatic acids	Substituted aromatics, aliphatics and carboxylic acids
LC 8	Hydroxy acids	Same as LC 7 plus hydroxy acids

*IR by grating spectrophotometer (nonvolatiles only analyzed).

†Site 302 had no IR data reported.

STUDY NUMBER 14

DATA
STATUS:

EVALUATION OF LEVEL 1 ORGANIC ANALYSIS SCHEMES

DATA
STATUS: Rough Draft, June 10, 1976

AUTHOR: P. Levins
CONTRACTOR: Arthur D. Little, Inc.
Contract No. 68-02-2150

PROJECT
OFFICER: L. D. Johnson
Process Measurements Branch
Industrial Environmental Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

The purpose of this analytical study was to identify problem areas in the Level I scheme and to address cost-effective alternatives. Two stated problem areas were the "less than optimum" LC fractionation scheme and the subsequent analysis of LC fractions exclusively by IR.

Several problems in the LC fractionation scheme from the *Technical Manual for Analysis of Organic Materials in Process Streams* were addressed. The lack of methodology for dealing with high molecular weight species was mentioned, and the use of thermal gravimetric analysis (TGA) to determine their presence was suggested. It was stated that samples containing high molecular weight species could be separated readily by gel permeation chromatography (GPC) procedures. A four-fraction LC scheme was suggested as more cost-effective than the eight-fraction scheme and as adequate for most environmental samples.

It was stated that IR analysis did not seem adequate to characterize complex environmental samples. LRMS analysis was suggested as an addition to the IR analysis for more complete identification of organic groups.

In order to compare the four-fraction LC scheme with the eight-fraction scheme, 12 environmental samples from several sources were supplied by PMB. Five samples were selected for fractionation by the eight-fraction scheme plus IR. Samples fractionated by the four-fraction scheme were analyzed by IR and LRMS.

GASEOUS GRAB

No sampling was performed in this study.

SASS

No sampling was performed in this study.

FUGITIVE EMISSIONS

No sampling was performed in this study.

LIQUIDS AND SLURRIES

No sampling was performed in this study.

SOLIDS

No sampling was performed in this study.

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TABLE 1. LC FRACTIONATION
SAMPLE 0018
EPA EIGHT-FRACTION SCHEME (E SERIES) vs. ADL FOUR-FRACTION SCHEME (A SERIES)
(wt. percent)

	A series				E series			
Fraction	TCO	Grav	Total	Total	TCO	Grav	Total	Total
LC 1		22				70		
LC 2		53				9		
LC 3		11				5		
LC 4		*				N.R.		
LC 5						N.R.		
LC 6						9		
LC 7						N.R.		
LC 8						N.R.		

*Not available because of initial blanks problem with aluminum pan.
N.R. = not reported.

TABLE 2. LC FRACTIONATION
SAMPLE 0053
EPA EIGHT-FRACTION SCHEME (E SERIES) vs. ADL FOUR-FRACTION SCHEME (A SERIES)
(wt. percent)

	A series				E series			
Fraction	TCO	Grav	Total	Total	TCO	Grav	Total	Total
LC 1		42				68		
LC 2		32				6		
LC 3		25				7		
LC 4		*				1		
LC 5						N.R.		
LC 6						12		
LC 7						N.R.		
LC 8						N.R.		

*Not available because of initial blanks problem with aluminum pan.
N.R. = not reported.

TABLE 3. LC FRACTIONATION
SAMPLE 0062
EPA EIGHT-FRACTION SCHEME (E SERIES) vs. ADL FOUR-FRACTION SCHEME (A SERIES)
(wt. percent)

Fraction	A series				E series			
	TCO	Grav	Total	Total	TCO	Grav	Total	Total
LC 1		39				76		
LC 2		36				3		
LC 3		9				1		
LC 4		*				N.R.		
LC 5						N.R.		
LC 6						2		
LC 7						N.R.		
LC 8						N.R.		

* Not available because of initial blanks problem with aluminum pan.
N.R. = not reported.

TABLE 4. LC FRACTIONATION
SAMPLE 0064
EPA EIGHT-FRACTION SCHEME (E SERIES) vs. ADL FOUR-FRACTION SCHEME (A SERIES)
(wt. percent)

Fraction	A series				E series			
	TCO	Grav	Total	Total	TCO	Grav	Total	Total
LC 1		0				16		
LC 2		57				4		
LC 3		38				15		
LC 4		5				13		
LC 5						5		
LC 6						32		
LC 7						2		
LC 8						N.R.		

N.R. = not reported.

TABLE 5. LC FRACTIONATION
SAMPLE 0065
EPA EIGHT-FRACTION SCHEME (E SERIES) VS. ADL FOUR-FRACTION SCHEME (A SERIES)
(wt. percent)

	A series				E series			
Fraction	TCO	Grav	Total	Total	TCO	Grav	Total	Total
LC 1		0				4		
LC 2		27				5		
LC 3		53				12		
LC 4		5				24		
LC 5						20		
LC 6						64		
LC 7						8		
LC 8						2		

TABLE 6. LC FRACTIONATION
SAMPLE 0066
EPA EIGHT-FRACTION SCHEME (E SERIES) VS. ADL FOUR-FRACTION SCHEME (A SERIES)
(wt percent)

	A series				E series			
Fraction	TCO	Grav	Total	Total	TCO	Grav	Total	Total
LC 1		1				43		
LC 2		57				9		
LC 3		21				6		
LC 4		2				7		
LC 5						4		
LC 6						12		
LC 7						1		
LC 8						1		

TABLE 7. IR REPORT
SAMPLE: NUMBER 18

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before LC separation	3,050	Weak	Unsaturated CH, probably aromatic
	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,600	Weak (broad)	Aromatic C=C
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3 bend
	725	Weak	CH_2 wag of more than four CH_2 groups
A1*	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3 bend
	725	Weak	CH_2 wag of more than four CH_2 groups
A2	3,450	Very weak (broad)	Bonded and unbonded OH (or NH)
	3,050	Weak to medium	Unsaturated CH, probably aromatic
	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,700	Very weak	C=0 of aryl aldehyde, acrylic ketone, aliphatic acid or α-β unsaturated acid
	1,600	Weak to medium (broad)	(1) Aromatic C=C; (2) NH bend; (3) COO^-; (4) chelated or conjugated carbonyl
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3
	1,095	Medium	C-0 of acyclic ether
	1,030	Weak	(1) Aromatic CH in-plane bend; (2) C-0 of alcohol
	870; 815; 750	Medium	Aromatic substitution bonds
	725	Medium	CH_2 wag of more than four CH_2 groups

*A1 to A4 is the ADL four-fraction scheme.

TABLE 7 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
A3	3,250	Weak (broad)	Bonded OH (or NH)
	3,050	Weak	Unsaturated CH, probably aromatic
	2,960; 2,930; 2,860	Medium	Saturated aliphatic CH_2, CH_3
	1,700	Weak to medium	Aliphatic acid or α-β unsaturated acid
	1,650	Weak (short)	(1) C=0 of amide; (2) C=C of olefin
	1,600	Medium (broad)	(1) Aromatic C=C; (2) NH bend; (3) COO^-; (4) chelated or conjugated carbonyl
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3 bend
	1,030	Weak	(1) Aromatic CH in plane bend (2) C-0 of alcohol
	870; 815; 750	Weak to medium	Aromatic substitution bands
	725	Weak	CH_2 wag of more than four CH_2 groups
A4	3,160	Weak	Bonded NH stretch
	3,055	Weak	Unsaturated CH, probably aromatic
	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,740	Very weak	C=0 of saturated aliphatic ester
	1,700	Very weak	C=0 of aryl aldehyde, acrylic ketone, aliphatic acid or α-β unsaturated acid
	1,640	Very weak	(1) C=0 of amide; (2) C=C of olefin
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3 bend
	1,410	Weak	CH_2 next to C=0
	1,020	Weak	(1) Aromatic CH in plane bend; (2) C-0 of alcohol
	870; 815; 750	Weak	Aromatic substitution bands
	725	Weak	CH_2 wag of more than four CH_2 groups

TABLE 7 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
E1*			Generally resembles spectra before separation, but with aromatic bands less strong.
E2	3,370	Weak to medium (broad)	Bonded OH (or NH)
	3,050	Weak (short)	Aromatic CH
	2,960; 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	2,730	Weak	Aldehyde CH
	1,735	Medium	Saturated ester
	1,715	Medium	(1) Unsaturated ester; (2) aldehyde
	1,600	Weak to medium	(1) Aromatic C=C; (2) chelated or conjugated carbonyl; (3) NH bend; (4) COO^-
	1,260	Weak	C-O of ester
	1,170; 1,130; 1,070; 1,030	Weak	C-O stretching of ethers, alcohols
	870; 815; 750	Weak to medium	Aromatic substitution bands
	725	Weak	CH_2 wag of more than four CH_2 groups

*E1 to E8 is EPA eight-fraction scheme as described in the Technical Manual for Analysis of Organic Materials in Process Streams.

TABLE 7 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
E3, 4, 5*			Generally similar with less aromatic content evident
	3,200-3,500	Weak (broad)	Bonded OH (or NH)
	3,050	Weak (short)	Aromatic CH
	2,960; 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	1,700	Weak (short)	C=0 of aldehyde, ketone
	1,600	Weak to medium	(1) Aromatic C=G; (2) chelated or conjugated carbonyl; (3) NH bend; (4) COO^-
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3
	1,030	Weak	(1) C-0 of alcohol; (2) aromatic CH in-plane bending
	870, 815, 750	Weak	Aromatic substitution bands
	725	Weak	CH_2 wag of more than four CH_2 groups
E6	3,250	Weak (broad)	Bonded OH (or NH)
	3,050	Very weak (short)	Aromatic CH
	2,960; 2,930; 2,860		Aliphatic CH_2, CH_3
	2,730	Weak	Aldehyde CH
	1,730	Weak to medium (short)	C=0 of saturated ester
	1,690	Medium (short)	C=0 of aldehyde or unsaturated ester
	1,650	Medium	(1) C=C of olefin; (2) C=N of imine; (3) C=0 of distributed amide
	1,600	Medium (broad)	(1) Aromatic C=C; (2) chelated or conjugated carbonyl; (3) NH bend; (4) COO^-
	1,030	Medium to strong	(1) C-0 of primary alcohol; (2) aliphatic aldehyde; (3) aromatic CH
	870; 815; 750	Weak to medium	Aromatic substitution bands
	725	Weak	CH_2 wag of more than four CH_2 groups

*E5 had very little sample and indicated mainly aliphatic hydrocarbon.

TABLE 7 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
E7	3,050	Very weak (short)	Aromatic CH
	2,960; 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	1,730	Weak (short, broad)	C=O of saturated ester
	1,600	Weak to medium (broad)	(1) Aromatic C=C; (2) chelated or conjugated carbonyl
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3
	1,100-1,300	Weak to medium (broad)	C-O stretching region
	1,030	Weak	Aromatic CH in-plane bending
	870; 815; 750	Weak	Aromatic substitution bonds
	725	Weak	CH_2 wag of more than four CH_2 groups

TABLE 8. IR REPORT
SAMPLE: NUMBER 53

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation	2,960; 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	1,720	Weak to medium	C=0 of aldehyde, ketone
	1,460; 1,380	Medium	CH_2, CH_3 bend
	1,000-1,150	Very weak (broad)	C-0 of ether or Si-0 or P-0
	725	Weak	CH_2 wag of mroe than four CH_2 groups
A1	3,080	Weak	Unsaturated CH, perhaps olefinic
	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,640	Very weak	C=C of olefin
	1,460; 1,380	Medium	CH_2, CH_3 bend
	970; 915	Weak	$CHR_1=CH_2$ type CH bending
	725	Weak	CH_2 wag of more than four CH_2 groups
A2	3,000-3,100	Weak	Unsaturated CH, probably aromatic
	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,730	Weak to medium	C=0 of saturated aldehyde or unsaturated ester or aryl ester
	1,600	Weak (broad)	(1) Aromatic C=C; (2) NH bend; (3) COO^-; (4) chelated or conjugated carbonyl
	1,460; 1,380	Medium	CH_2, CH_3 bend
	1,120	Weak	C-0 of aromatic ester
	1,070; 1,030	Weak	(1) C-0 of aromatic ether (2) aromatic CH in plane bend; (3) Si-0 (impurity?)
	870	Weak	Aromatic substitution patterns: 1,3; 1,3,5; 1,2,3,5; 1,2,4,5; 1,2,3,4,5
	815	Weak	Aromatic substitution patterns: 1,4-; 1,2,3,4; 1,2,4; 1,3,5
	750	Weak	Aromatic substitution patterns: 1,2-; 1,3
	725	Weak	CH_2 wag of more than four CH_2 groups

TABLE 8 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
A3	3,100-3,600	Weak to medium (very broad)	Bonded and unbonded OH (or NH)
	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH$_2$, CH$_3$ stretch
	1,770	Weak (short)	C=0 of acid halide, lactone, or vinylic or phenolic ester
	1,750	Weak to medium (short)	C=0 of saturated ester or α-keto ester or α-diester
	1,710	Strong	C=0 of aryl aldehyde, acyclic ketone, aliphatic acid or α-β-unsaturated acid
	1,610	Weak to medium	(1) Aromatic C=C; (2) NH bend; (3) COO$^-$; (4) chelated or conjugated carbonyl
	1,460; 1,380	Medium	Aliphatic CH$_2$, CH$_3$ bend
	1,410	Weak	(1) CH$_2$ next to C=0 (2) COO$^-$
	1,260; 1,165	Weak	Aldehyde or ketone skeletal vibrations
	1,120	Weak	C-0 of aromatic ester
	1,020	Weak	(1) C-0 of alcohol; (2) Si-0, P-0; (3) aromatic CH in-plane bend
	725	Weak	CH$_2$ wag of more than four CH$_2$ groups

TABLE 8 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
A4	3,300-3,600	Weak (broad)	Bonded and unbonded OH (or NH)
	3,150	Weak	NH stretch
	3,060	Weak	Unsaturated CH, probably aromatic
	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,780	Weak (short)	C=0 of acid halide, lactone, or vinylic or phenolic ester
	1,740	Weak (short)	C=0 of saturated ester
	1,715	Medium	C=0 of aryl aldehyde, acyclic ketone, aliphatic acid or α-β unsaturated acid
	1,595	Medium	(1) Aromatic C=C; (2) chelated carbonyl; (3) NH bend; (4) COO
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3
	1,410	Weak	(1) CH_2 next to C=0; (2) COO^-
	1,260	Weak to medium	Aldehyde or ketone skeletal vibrations
	1,170	Weak to medium	Aldehyde or ketone skeletal vibrations
	1,120	Weak to medium	C-0 of aromatic ester
	1,020	Weak to medium	(1)C-0 of alcohol; (2) Si-0, P-0 (3) aromatic CH in-plane bend
	725	Weak to medium	CH_2 wag of more than four CH_2 groups
E1			Curve indicates aliphatic hydrocarbons only

TABLE 8 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
E2		(Very weak aromatic indications)	
	3,200-3,500	Very weak (broad)	Bonded OH (or NH)
	2,960; 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	2,730	Weak	Aldehyde CH
	1,720	Medium to strong	Unsaturated or aromatic ester C=0
	1,700	Medium	C=0 of aldehyde or ketone
	1,605	Weak	(1) Aromatic C=C; (2) chelated or conjugated carbonyl; (3) NH bend
	1,125	Medium	(1) C-0 of aromatic ester; (2) aldehyde, ketone skeletal vibration
	1,070; 1,030	Medium	(1) C-0 of alcohol or ether; (2) aldehyde skeletal vibration; (3) Si-0
	725	Weak	CH_2 wag of more than four CH_2 groups
E3,4,5			All similar to E2
E6		(Stronger carbonyl fraction)	
	2,500-3,600	Weak (broad)	Strongly bonded OH (or NH)
	2,960; 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	1,770	Weak (short)	C=0 of acid halide or lactone
	1,710	Short (broad)	C=0 of acid, aldehyde or ketone
	1,610	Weak (short)	(1) Chelated or conjugated carbonyl; (2) NH bend; (3) COO^-
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3
	1,410	Very weak (short)	(1) CH_2 next to C=0; (2) COO^-
	1,100-1,300	Weak to medium (broad)	C-0 stretching of ether, alcohol, ester
	950-1,050	Weak to medium (broad)	C-0 stretching of ether, alcohol, ester
	725	Weak	CH_2 wag of more than four CH_2 groups

TABLE 8 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
E7			Used entire sample. Curve generally similar to E6 but broader peaks at 1,600 cm^{-1} and 1,400 cm^{-1} (COO^-). Also new peaks at 1,055 cm^{-1} (?) and 760, 790 cm^{-1} (C-Cl bands?). A peak at 1,560 cm^{-1} may be due to nitro groups.
E8			Very little sample. Curve indicated some aliphatic hydrocarbon and a very broad 1,550-1,650 cm^{-1} band.

TABLE 9. IR REPORT
SAMPLE: NUMBER 64

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation	3,530	Weak	Unbonded OH
	3,350	Weak (broad)	Bonded OH (or NH), phenols
	3,030; 3,050	Weak to medium	Unsaturated CH, probably aromatic
	2,960, 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	2,400-2,700	Weak (broad)	Bonded OH
	1,600	Strong	(1) Chelated or conjugated carbonyl =C-C=0, e.g., acetylacetone, hydroxyacetophenone; (2) aromatic C=C with -0- substituent; (3) COO^-
	1,480-1,550	Medium (short)	Aromatic ring stretching
	1,450	Medium to short	(1) Aliphatic CH_2, CH_3; (2) aromatic C=C; (3) ionic carbonate
	1,375	Medium	CH_3 groups
	1,260	Weak to medium	(1) aryl and aralkyl ethers =C-0 (phenoxy) (2) alkyl ketones
	1,110-1,350	Medium (broad)	Aryl and aralkyl ethers =C-0 (phenoxy)
	960; 1,035	Weak	(1) Clay minerals; (2) phenoxy; (3) aromatic CCH in single or condensed rings
	870	Medium to strong	1,3-disubstitution aromatic
	855	Weak to medium	β-naphthalenes
	815	Medium to strong	1,4-; 1,2,4-; 1,3,5-; β-naphthalenes
	740	Medium to strong	1,2-disubstitution
	705	Weak	Mono- or 1,3-disubstitution

TABLE 9 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
A1			Very little sample, aliphatic hydrocarbon present.
A2			Resembles total sample, with broad hydroxyl band narrowed and centered at 3,430 cm^{-1}. Two aromatic bands appeared stronger; 705 cm^{-1} and 855 cm^{-1}.
A3			Resembles total sample.
A4			Very little sample, but generally resemble total.
E1			Curve generally resembles total sample with very little hydroxyl evident, and C-O bands more defined.
E2			Very similar to E1 with aromatic bands at 710 cm^{-1} and 840 cm^{-1} appearing stronger.
E3			Similar to total, but containing two well-defined hydroxyl bands at 3,540 cm^{-1} (unbonded OH) and 3,425 cm^{-1} (more bonded OH or NH).
E4			Resembles total sample with extra shoulder at 1,700 cm^{-1} (acid, ketone, or aldehyde carbonyl.
E5			Resembles E4 with broader hydroxyl band
E6			Resembles total sample.
E7			Very little sample but resembles total.

TABLE 10. IR REPORT
SAMPLE: NUMBER 66

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation	3,550	Weak	Unbonded OH or NH
	3,430	Weak	Bonded OH or NH
	3,060*	Medium	Unsaturated CH, probably aromatic
	2,960; 2,930; 2,860	Strong	Aliphatic CH_2, CH_3
	1,600	Strong	(1) Chelated or conjugated carbonyl =C-C=0; (2) aromatic C=C with -0- substituent; (3) COO^-
	1,500	Medium (short)	Aromatic ring structure
	1,450	Medium to short	(1) Aliphatic CH_2, CH_3 (2) aromatic C=C; (3) ionic carbonate
	1,380	Medium	CH_3 groups
	1,330; 1,300; 1,275; 1,190	Weak (broad)	Aryl and aralkyl ethers = C-0-(phenoxy)
	1,240*; 1,140*; 1,040; 1,010; 950	Weak to medium	(1) Aromatic CCH in single or condensed rings; (2) clay minerals; (3) phenoxy
	865	Weak to medium	*1,3 disubstituted aromatic
	845	Weak to medium	*β-naphthalenes
	815	Strong	*1,4-; 1,2,4-; 1,3,5-; β-naphthalenes
	740	Strong	*1,2 disubstituted
	715	Weak to medium	*Mono or 1,3 - disubstituted
	620	Weak to medium	*?
A1			Curve resembles aliphatic hydrocarbon only.

*Present in phenanthrene.

TABLE 10 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
A2			Curve has more definition with stronger aromatic character, but generally resembles total (broad C-0 ether band not present).
A3			Curve generally resembles 64-A3 with more definition in 1,150-1,300 cm^{-1} region and slight shift in aromatic substitution bands pattern.
A4			Less sample, but similar to A3
E1			Resembles total sample; generally, all bands are more well-defined; 1,600 cm^{-1} band more resolved with shoulders on either side; hydroxyl bands at 3,430 cm^{-1} and 3,550 cm^{-1} have disappeared; broad absorption 1,150-1,300 (C-0 of phenolics) not evident
E2			Resembles E1; aliphatic character greatly reduced.
E3			Aromatic substitution pattern changed (810, 750, 730); aliphatic character stronger than E2; hydroxyl bands at 3,430 cm^{-1} (bonded OH) very strong with weak band at 3,550 cm^{-1} (bonded OH), with corresponding phenolic C-0 bands at 1,240 and 1,330 cm^{-1}

TABLE 10 (con.)

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
E4			Resembles 64-E5 sample with addition of weak carbonyl at 1,705 cm^{-1}, bands at 850 cm^{-1} and 1,005 cm^{-1}, and much stronger and broader hydroxyl.
E5			Resembles 64-E5 with stronger aromatic definition and broad phenolic C-O centered at 1,200 cm^{-1} rather than 1,250 cm^{-1}.
E6			Well-defined aromatic character lost; broad OH band from 2,300-3,500 cm^{-1} (bonded hydroxyl); methyl to methylene ratio higher.
E7			Very little sample; only aliphatic CH_2 and CH_3 evident.
E8			Very little sample; generally resembles E6.

TABLE 11. IR REPORT
SAMPLE: NUMBER 48

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
A6,8			All contained bands indicating aliphatic hydrocarbon and weak ester carbonyl (plus silicone oil impurity).

TABLE 12. IR REPORT
SAMPLE: NUMBER 55

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation			All contained bands indicating aliphatic hydrocarbon and weak ester carbonyl (plus silicone oil impurity). A Soxhlet blank also showed aliphatic hydrocarbon, weak (aldehyde or unsaturated ester) carbonyl, and silicone oil impurity.

TABLE 13. IR REPORT
SAMPLE: NUMBER 61

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation	2,960; 2,930; 2,860	Strong	Saturated aliphatic CH_2, CH_3
	1,600	Very weak	(1) C=C of aromatic ring; (2) chelated or conjugated carbonyl; (3) COO
	1,460; 1,380	Medium	Aliphatic CH_2, CH_3
	1,050-1,200	Very weak (broad)	C-0 stretching region
	725	Weak	CH_2 wag of more than four CH groups

TABLE 14. IR REPORT
SAMPLE: NUMBER 62

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation			Resembles sample 64 with much stronger aliphatic content, very weak hydroxyl, and absence of broad "phenoxy" band from 1,100-1,350 cm^{-1}.

TABLE 15. IR REPORT
SAMPLE: NUMBER 63

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation			Resembles sample 62

TABLE 16. IR REPORT
SAMPLE: NUMBER 65

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation			Resembles sample 64.

TABLE 17. IR REPORT
SAMPLE: NUMBER 67

Sample description	Wave number (cm^{-1})	Intensity	Assignment (comments)
Before separation	,		Resembles sample 66 with less aliphatic content and stronger band of 780 cm^{-1} (probably 1,3- or 1,2,3- aromatic substitution).

TABLE 18. LRMS REPORT

Fraction	MS observations (in order of intensity)
Sample 0018	
Total	Very complex, to m/e 1,200, principally saturated aliphatic. More than 75% vaporized.
A-1	Aliphatics; 2n, 2n-2, 2n-4, and 2n-6 all about equal. 2n-6 and 2n-8 predominate at high temperature. 75% vaporized.
A-2	Complex, to m/e >1,000; substituted naphthalenes and phenanthrenes present. 50% vaporized.
A-3	Complex, to m/e >1,000; palmitic acid, phthalate, stearic acid. 50% vaporized.
A-4	Complex, to m/e ~750; small amount of substituted naphthalenes. <50% vaporized.
Sample 0053	
Total	Very complex, to m/e >1,000, mostly saturated. 50% vaporized.
A-1	Aliphatics, 25% 2n, 19% 2n+2, 19% 2n-2, 16% 2n-6, 13% 2n-4, 7% 2n-8. Completely vaporized.
A-2	Aromatics and substituted aromatics, in order of abundance: 2n-6, 2n-8, 2n-10, 2n-12, 2n-14; also some nitrogenous material. 50% to 75% vaporized.
A-3	Very complex, to m/e 300. Less than 50% vaporized.
A-4	In order of abundance: palmitic acid, stearic acid, methyl palmitate, methyl stearate. 50% to 75% vaporized.

TABLE 18 (con.)

Fraction	MS observations (in order of intensity)
Sample 0055	
Total extract	Dioctylphthalate; 75% vaporized.
Sample 0061	
Total	Complex, in order of abundance: aliphatics to m/e 350; alkyl benzenes; nitrogenous material; alkyl naphthalenes. 50% vaporized.
Sample 0062	
Total	Aliphatics predominate to m/e 1,100, mostly saturated.
A-1	Aliphatics to m/e 1,150, in order of abundance: 2n+2, 2n, 2n-6, 2n-2, 2n-8, 2n-10. 75% vaporized.
A-2	Alkyl benzenes, to C_7 Φ; aliphatics to m/e >960; N species; 2n-16 series. 50% vaporized.
A-3	Complex, to m/e ~ 1,000; contains some phthalates and fatty acids. 50% vaporized.
A-4	Very little to be seen; some palmitic and stearic acids. 50% to 75% vaporized.
Sample 0063	
Total	Complex, to m/e >1,200; aliphatics, alkyl benzenes to C_7Φ; alkyl naphthalenes; some PNA material; carbazole. 75% vaporized.

TABLE 18 (con.)

Fraction	MS observations (in order of intensity)
Sample 0066	
Total	Anthracene, methyl anthracene, other substituted anthracenes, pyrene, carbazole, methyl pyrene, other PNA's to C_{20}^{+}. 50% to 75% vaporized.
A-1	Paraffins, to C_{30} and beyond; C_{18} to C_{23} the largest. ~90% vaporized.
A-2	Anthracene, methylanthracene, chrysene, methyl chrysene, dimethyl chrysene and many combinations of substituted PNA's. 75% to 90% vaporized.
A-3	Methyl benzthiophene, ethyl benzaldehyde, methyl quinoline, benzylphenol, dimethylbenzthiophene, phenyl phenol, ethyl quinoline, methyl cresol, etc. 50% to 75% vaporized.
A-4	Very little; complex to m/e 400.
Sample 0067	
Total	Anthracene, pyrene, methyl anthracene, methyl pyrene, other substituted PNA;s, chrysene, carbazole, benzpyrene. 50% to 75% vaporized.
Other fractions	
6	Polyphenyl ether; silica gel residue.
8	Polyphenyl ether; silica gel residue.

TABLE 19. LRMS REPORT
COMPOUNDS MOST PROBABLY PRESENT
SAMPLE 0066

Percent of total sample	
Aromatics	
23	Anthracene/phenanthrene
6	Pyrene
6	Methyl anthracene
5	Diphenyl ethane/hydroxy fluorene
4	Stilbene
3	Diphenyl propane
3	Methyl pyrene
2	Fluorene
2	Methyl phenyl benzene
2	Methyl stilbene/diphenyl propene
2	Phenyl naphthalene
2	Dimethyl phenanthrene
2	Hexadecahydro pyrene
2	Trimethyl phenanthrene
1	Chrysene
5	Others, unidentified but above 1% level
Oxygenates	
4	Methoxy biphenyl/diphenylene propene
2	Methoxy stilbene
5	Hydroxyfluorene/diphenyl ethane
Thiophenes	
2	Methyl dibenzthiophene
1	Dimethylnaphthothiophene

TABLE 20. LRMS REPORT
COMPOUNDS MOST PROBABLY PRESENT
SAMPLE FRACTION 66 A-3

Percent of total sample	
Oxygenates	
4	Benzylphenol
4	Methoxyfluorene
3	Ethylbenzaldehyde/terphthaldehyde
3	Phenyl phenol
3	Hydroxyfluorene
3	Methoxystilbene
3	Dimethoxy biphenyl
2	Hydroxybenzaldehyde
2	Terephthalic acid/piperonal
2	Cyclohexylphenol
1	Cresol
1	Methoxybenzaldehyde
Thiophenes	
5	Methylbenzthiophene/methoxy propyl benzene
5	Methyldibenzthiophene
4	Dimethylbenzthiophene
3	Dimethylnaphthothiophene
Nitrogenous	
5	Methylbenzoquinoline
4	Methylquinoline
4	Ethyl quinoline
4	Benzo quinoline/acridine
3	Methyl phenyl indole

STUDY NUMBER 15

DATA SOURCE:

EVALUATION OF SELECTED METHODS FOR CHEMICAL AND BIOLOGICAL TESTING OF INDUSTRIAL PARTICULATE EMISSIONS

EPA-600/2-76-137

DATA STATUS:

Final Report, May 1976

AUTHOR:
CONTRACTOR:

H. Mahar
The Mitre Corporation
Westgate Research Park
McLean, Virginia 22101
Contract No. 68-02-1859, Task 5
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Program Element No. 1AB013

PROJECT OFFICER:

L. D. Johnson
Process Measurements Branch
Industrial Environmental Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

This report presents results of chemical analyses (SSMS, GC-MS, HRMS) and cellular biological assays (RAM Cytotoxicity, Ames') performed on size-classified particulates collected at nine industrial sites using a SASS train. The industrial processes sampled included the following:

1. Steel plant open hearth furnace,
2. Steel plant coke oven heater,
3. Steel plant basic oxygen furnace,
4. Steel plant iron sintering,
5. Oil fired power plant,
6. Copper smelting,
7. Aluminum smelter,
8. Ceramics plant,
9. Sludge incinerator, and
10. Pulp and paper mill.

The purpose of the study was to evaluate the effectiveness of selected testing methods in accomplishing the following objectives.

1. Obtain size-classified particulate samples,
2. Obtain toxicity ratings of samples,
3. Obtain mutagenic ratings of samples, and
4. Correlate chemical and biological data.

Results indicate that objectives one, two, and three can be effectively accomplished; however, no strong correlation between the chemical and biological results was established.

With a target of 300 mg of particulate to collect, each run was successful except the 3μ-10μ cyclone collection at the open hearth furnace, coke oven heater, and the basic oxygen furnace. The oil-fired power plant shut down 2 hours after sampling began resulting in <300 mg. The pulp and paper mill substantially reduced the stack emissions during the sampling as compared to the previous day and resulted in a negligible amount of particulate being collected. Analyses of this site were therefore not conducted. Particulates >10μ were not analyzed due to their "minor air pollution hazard," based on their short airborne time.

Results of the study indicate that the SASS train operated successfully under a variety of circumstances, but was quite awkward and difficult to manage. The particulate sizes (1μ-3μ vs. 3μ-10μ) produced a substantial variation in results of elemental composition as well as GC-MS, cytotoxicity, and mutagenic activity. Arsenic concentrations were highest in copper smelting samples. Polycyclic organic species were detected only in the 1μ-3μ sample from the iron sintering plant.

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The concentrations of coronene and dibenzocarbazole, as determined by GC-MS and HRMS analysis of aluminum smelter samples, indicate HRMS produced results ranging two to seven times greater than those obtained by GC-MS. Bioassay results on the aluminum smelter show a weak mutagenic response in the 1μ-3μ particulates while the copper smelter 1μ-3μ particulates indicated possible mutagenic activity.

GASEOUS GRAB

Sampling was not performed in this study.

SASS

Sampling locations and conditions are listed in Table 1 with an illustration in Figure 1. Stainless steel CRES 316 was used exclusively for the cyclones, tubing, and fitting; Viton "O" rings were used as seals and Teflon needle felt material was used as the filter material. At 3 scfm, cyclone cutoffs were 9.5μ, 2.0μ, and 0.5μ. Sampling was limited to a maximum of 5 hours (intermittently, if particulate loading was high). No impingers or organic collection module were used.

FUGITIVE EMISSIONS

Sampling was not performed in this study.

LIQUIDS AND SLURRIES

Sampling was not performed in this study.

SOLIDS

Sampling was not performed in this study.

TABLE 1. LOGISTICS OF SAMPLE COLLECTION

Source	Open Hearth Furnace	Coke Oven Heater	Basic Oxygen Furnace	Iron Sintering Plant	Oil Fired Power Plant	Clay Aggregate Plant	Copper Smelter	Aluminum Smelter	Minicipal Sludge Incinerator	Kraft Mill Process
Sampling location	Electrostatic precipitator (4 duct diameters downstream from ESP, 4 duct diameters upstream of stack)	Base of stack	Downstream of ESP, downstream of induction fan, upstream of stack	Inlet to baghouse	Wet scrubber inlet	Between primary and secondary cyclones	Outlet of roaster reverberator inlet to baghouse	Inlet to baghouse	Duct between furnace and water quench	Stack effluent from ESP
T° stack*	350°-425°	400°	150-225°	400°	170°	510°	250°	210°	1100°	335°
T° 10μ cyclone*	350°	380°	270°	350°	195°	400°	275°	300°	410°	335°
T° oven*	350°-400°	380°-400°	270°	390°	250°	400°	300°	300°	380°	350°
Flow rate through sampling train	4.8 scfm	3.5 scfm	4.6 scfm	5 scfm	4.5 scfm	3.8 scfm	4.7 scfm	5 scfm	4.7 scfm	4.8 scfm
Total sampling time	5 hours continuous	5 hours continuous	5 hours continuous	2 hours intermittent over 5 hour period	2 hours continuous	1.25 hours intermittent over 5 hour period	1 hour continuous	2 hours intermittent over 5 hour period	5 hours continuous	5 hours continuous

*Degrees fahrenheit

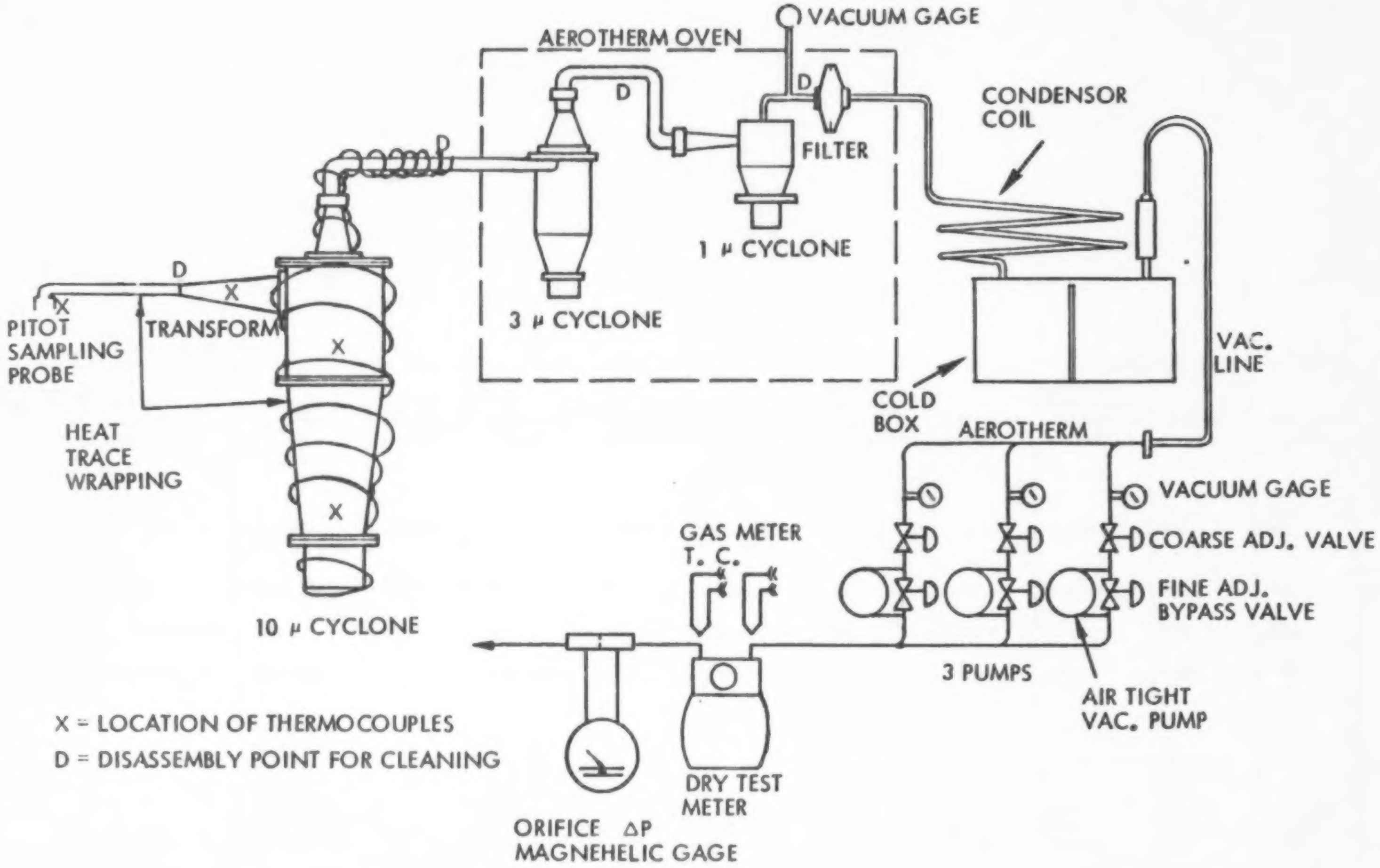
VACUUM GAGE
AEROTHERM OVEN
D
D
D
CONDENSOR COIL
FILTER
1 μ CYCLONE
D
X
PITOT SAMPLING PROBE
TRANSFORM
X
3 μ CYCLONE
VAC. LINE
COLD BOX
AEROTHERM
HEAT TRACE WRAPPING
X
VACUUM GAGE
GAS METER T. C.
COARSE ADJ. VALVE
FINE ADJ. BYPASS VALVE
10 μ CYCLONE
3 PUMPS
AIR TIGHT VAC. PUMP
X = LOCATION OF THERMOCOUPLES
D = DISASSEMBLY POINT FOR CLEANING
DRY TEST METER
ORIFICE ΔP MAGNEHELIC GAGE

Figure 1. Series cyclone train, field sampling configuration.

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY
1μ-3μ PARTICULATES
(ppm)

U	1.5	Dy		Rh		Cr	60
Th	<0.22	Tb		Ru		V	330
Bi	21	Gd		Mo	79	Ti	15
Pb	≅1,900	Eu		Nb	0.9	Sc	<0.16
Tl	1.5	Sm		Zr	1.4	Ca	0.83%
Hg		Nd	0.65	Y	0.51	K	3.0%
Au		Pr	0.13	Sr	11	Cl	260
Pt		Ce	1.1	Rb	95	S	4.0%
Ir		La	0.20	Br	43	P	≅4,900
Os		Ba	12	Se	2.3	Si	≅4,300
Re	*	Cs	2.5	As	480	Al	140
W	10	I	3.9	Ge	18	Mg	1.0%
Ta	0.79	Te	0.40	Ga	87	Na	8.0%
Hf		Sb	70	Zn	2.0%	F	760
Lu		Sn	610	Cu	≅2,900	B	31
Yb		In	*	Ni	470	Be	0.34
Tm		Cd	44	Co	110	Li	110
Er		Ag	65	Fe	40%		
Ho		Pd		Mn	≅4,400		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
STEEL PLANT OPEN HEARTH FURNACE
3μ-10μ PARTICULATES
(ppm)

U	0.33	Dy		Rh		Cr	880
Th	<0.23	Tb		Ru		V	290
Bi	11	Gd		Mo	35	Ti	33
Pb	900	Eu		Nb	0.93	Sc	<0.16
Tl	0.53	Sm		Zr	0.64	Ca	2.0%
Hg		Nd	0.68	Y	0.78	K	0.69%
Au		Pr	0.13	Sr	4.8	Cl	270
Pt		Ce	1.2	Rb	32	S	2.0%
Ir		La	0.21	Br	10	P	0.5%
Os		Ba	6.3	Se	2.7	Si	0.89%
Re	*	Cs	0.26	As	190	Al	68
W	11	I	1.7	Ge	7.1	Mg	1.0%
Ta	0.82	Te	0.15	Ga	90	Na	2.0%
Hf		Sb	27	Zn	2.0%	F	790
Lu		Sn	340	Cu	≅3,600	B	47
Yb		In	*	Ni	490	Be	<0.24
Tm		Cd	5.4	Co	120	Li	59
Er		Ag	41	Fe	40%		
Ho		Pd		Mn	0.52%		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
STEEL PLANT BASIC OXYGEN FURNACE
1μ-3μ PARTICULATES
(ppm)

U	<0.14	Dy		Rh		Cr	170
Th	<0.14	Tb		Ru		V	23
Bi	2.9	Gd		Mo	10	Ti	9.6
Pb	210	Eu		Nb	2.7	Sc	<0.10
Tl	0.97	Sm		Zr	0.40	Ca	2.0%
Hg		Nd	0.18	Y	0.71	K	2.0%
Au		Pr	0.04	Sr	14	Cl	3.0%
Pt		Ce	0.73	Rb	74	S	5.0%
Ir		La	0.28	Br	65	P	≅1,200
Os		Ba	40	Se	0.40	Si	0.56%
Re	*	Cs	1.2	As	52	Al	21
W	6.8	I	2.6	Ge	6.0	Mg	≅2,500
Ta	0.52	Te	<0.08	Ga	57	Na	.077%
Hf		Sb	9.7	Zn	≅4,600	F	6.0%
Lu		Sn	40	Cu	800	B	50
Yb		In	*	Ni	46	Be	<0.15
Tm		Cd	12	Co	57	Li	16
Er		Ag	43	Fe	40%		
Ho		Pd		Mn	0.77%		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY
STEEL PLANT BASIC OXYGEN FURNACE
3μ-10μ PARTICULATES
(ppm)

U	6.2	Dy		Rh		Cr	220
Th	<0.25	Tb		Ru		V	40
Bi	5.0	Gd		Mo	38	Ti	0.44
Pb	170	Eu		Nb	2.3	Sc	<0.18
Tl	0.78	Sm		Zr	0.69	Ca	4.0%
Hg		Nd	0.73	Y	0.56	K	4.0%
Au		Pr	0.15	Sr	43	Cl	3.0%
Pt		Ce	5.9	Rb	63	S	≅3,500
Ir		La	2.2	Br	110	P	≅3,500
Os		Ba	68	Se	0.51	Si	1.0%
Re	*	Cs	2.8	As	67	Al	73
W	12	I	19	Ge	10	Mg	2.0%
Ta	0.44	Te	0.15	Ga	97	Na	9.0%
Hf		Sb	17	Zn	0.58%	F	5.0%
Lu		Sn	140	Cu	1,100	B	130
Yb		In	*	Ni	93	Be	<0.26
Tm		Cd	9.7	Co	130	Li	27
Er		Ag	440	Fe	>80%		
Ho		Pd		Mn	1.0%		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY
STEEL PLANT IRON SINTERING
1μ-3μ PARTICULATES
(ppm)

U	3.7	Dy	1.3	Rh		Cr	52
Th	3.7	Tb	0.15	Ru		V	33
Bi	100	Gd	0.28	Mo	17	Ti	370
Pb	0.82%	Eu	0.67	Nb	4.5	Sc	1.1
Tl	35	Sm		Zr	14	Ca	30%
Hg		Nd	6.5	Y	25	K	3.0%
Au		Pr	2.8	Sr	110	Cl	1.0%
Pt		Ce	46	Rb	≅950	S	1.0%
Ir		La	10	Br	270	P	890
Os		Ba	140	Se	310	Si	2.0%
Re	*	Cs	25	As	120	Al	890
W	3.6	I	69	Ge	9.2	Mg	4.0%
Ta	0.20	Te	2.0	Ga	4.4	Na	5.0%
Hf	0.44	Sb	3.5	Zn	≅1,100	F	0.7%
Lu	0.24	Sn	6.1	Cu	≅2,200	B	76
Yb	0.21	In	*	Ni	64	Be	0.39
Tm	0.09	Cd	22	Co	59	Li	28
Er	0.37	Ag	35	Fe	2.0%		
Ho	0.09	Pd		Mn	321		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY
STEEL PLANT IRON SINTERING
3μ-10μ PARTICULATES
(ppm)

U	2.3	Dy	1.8	Rh		Cr	76
Th	2.3	Tb	0.10	Ru		V	48
Bi	5.7	Gd	0.41	Mo	11	Ti	290
Pb	≅2,100	Eu	0.98	Nb	6.5	Sc	1.1
Tl	11	Sm	1.1	Zr	10	Ca	40%
Hg		Nd	9.5	Y	65	K	1.0%
Au		Pr	0.95	Sr	160	Cl	≅3,300
Pt		Ce	38	Rb	180	S	0.31%
Ir		La	7.3	Br	150	P	1,300
Os		Ba	60	Se	89	Si	3.0%
Re	*	Cs	18	As	35	Al	≅1,300
W	6.8	I	29	Ge	2.9	Mg	6.0%
Ta	0.29	Te	0.58	Ga	6.3	Na	3.0%
Hf	0.63	Sb	2.5	Zn	760	F	1.0%
Lu	0.06	Sn	4.4	Cu	≅1,000	B	61
Yb	0.57	In	*	Ni	26	Be	0.57
Tm	0.03	Cd	14	Co	86	Li	18
Er	0.53	Ag	6.1	Fe	6.0%		
Ho	0.13	Pd		Mn	640		

*Internal standard.

No data indicates "below detective limit of 0.1 ppmw."

TABLE 8. SPARK SOURCE MASS SPECTROSCOPY†
COPPER SMELTING
1μ-3μ PARTICULATES
(ppm)

U	5.6	Dy		Rh		Cr	190 (153)
Th	0.56	Tb		Ru		V	49 (128)
Bi	2.0% (5,700)	Gd		Mo	0.51% (570)	Ti	560 (170)
Pb	35% (11.2%)	Eu		Nb	31 (5.3)	Sc	0.78 (<15)
Tl	≅3,900 (875)	Sm		Zr	11 (9.8)	Ca	0.75% (800)
Hg	(Trace)	Nd	9.8	Y	19 (16)	K	10% (1,600)
Au	25	Pr	2.0	Sr	29 (45)	Cl	610 (135)
Pt		Ce	3.9	Rb	140 (105)	S	2.0%
Ir		La	4.1	Br	750 (52)	P	≅1,300 (180)
Os		Ba	170 (31)	Se	0.55% (865)	Si	1.0% (3,000)
Re		Cs	1.9 (60)	As	3.0% (21%)	Al	250 (940)
W	180	I	160 (25)	Ge	140 (120)	Mg	2.0% (1.1%)
Ta	1.3	Te	≅3,600 (1,700)	Ga	6.5 (18)	Na	3.0% (1,700)
Hf		Sb	8.0% (5.8%)	Zn	1.0% (1.7%)	F	210
Lu		Sn	4.0% (3,700)	Cu	5.0% (2.9%)	B	11
Yb		In	*	Ni	1,300	Be	5.9
Tm		Cd	0.49% (3,800)	Co	180 (69)	Li	43
Er		Ag	≅1,100 (420)	Fe	3.0% (1.0%)		
Ho		Pd		Mn	310 (73)		

*Internal standard.

†Data in parentheses generated by EMSL/RTP.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 9. SPARK SOURCE MASS SPECTROSCOPY
COPPER SMELTING
3μ-10μ PARTICULATES
(ppm)

U	2.4	Dy		Rh		Cr	79
Th	1.1	Tb		Ru		V	98
Bi	1.0%	Gd	0.31	Mo	≅2,500	Ti	≅1,100
Pb	4.0%	Eu		Nb	6.7	Sc	1.7
Tl	1,100	Sm	3.6	Zr	11	Ca	4.0%
Hg	(Trace)	Nd	9.8	Y	8.1	K	1.0%
Au		Pr	2.0	Sr	82	Cl	≅1,100
Pt		Ce	20	Rb	43	S	2.0%
Ir		La	7.9	Br	750	P	≅1,300
Os		Ba	81	Se	≅3,400	Si	3.0%
Re	*	Cs	3.7	As	6.0	Al	≅2,500
W	13	I	160	Ge	24	Mg	1.0%
Ta	79	Te	≅1,300	Ga	13	Na	7.0%
Hf		Sb	3.0%	Zn	0.7%	F	370
Lu		Sn	≅4,600	Cu	20%	B	23
Yb		In	*	Ni	760	Be	59
Tm		Cd	≅1,400	Co	480	Li	18
Er		Ag	≅2,200	Fe	20%		
Ho		Pd		Mn	880		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 10. SPARK SOURCE MASS SPECTROSCOPY
ALUMINUM SMELTER
1μ-3μ PARTICULATES
(ppm)

U	1.3	Dy		Rh		Cr	90
Th	<0.24	Tb		Ru		V	0.51%
Bi	270	Gd		Mo	67	Ti	130
Pb	330	Eu		Nb	1.8	Sc	<0.17
Tl	3.0	Sm		Zr	5.3	Ca	≅1,800
Hg		Nd	1.1	Y	2.2	K	≅4,100
Au		Pr	0.08	Sr	9.4	Cl	690
Pt		Ce	0.45	Rb	98	S	3.0%
Ir		La	0.37	Br	86	P	0.76%
Os		Ba	5.2	Se	22	Si	740
Re	*	Cs	7.5	As	≅4,100	Al	2.0%
W	21	I	34	Ge	3.4	Mg	330
Ta	1.5	Te	26	Ga	900	Na	8.0%
Hf		Sb	130	Zn	160	F	>50%
Lu		Sn	100	Cu	≅2,200	B	72
Yb		In	*	Ni	≅2,800	Be	2.9
Tm		Cd	16	Co	43	Li	9.8
Er		Ag	2.0	Fe	4.0%		
Ho		Pd		Mn	20		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 11. SPARK SOURCE MASS SPECTROSCOPY
ALUMINUM SMELTER
3μ-10μ PARTICULATES
(ppm)

U	0.40	Dy		Rh		Cr	56
Th	<0.12	Tb		Ru		V	≅1,500
Bi	56	Gd		Mo	51	Ti	140
Pb	78	Eu		Nb	0.48	Sc	<0.08
Tl	0.19	Sm		Zr	1.5	Ca	2.0%
Hg		Nd		Y	1.4	K	840
Au		Pr	0.03	Sr	50	Cl	140
Pt		Ce	0.14	Rb	6.1	S	0.6%
Ir		La	0.05	Br	23	P	≅2,000
Os		Ba	25	Se	14	Si	230
Re	*	Cs	0.27	As	700	Al	≅3,500
W	20	I	16	Ge	2.1	Mg	870
Ta	0.91	Te	4.3	Ga	200	Na	27
Hf		Sb	66	Zn	56	F	20%
Lu		Sn	33	Cu	≅4,600	B	27
Yb		In	*	Ni	≅3,800	Be	1.8
Tm		Cd	1.8	Co	27	Li	13
Er		Ag	1.1	Fe	2.0%		
Ho		Pd		Mn	6.3		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 12. SPARK SOURCE MASS SPECTROSCOPY
CERAMICS PLANT
1μ-3μ PARTICULATES
(ppm)

U	10	Dy	1.7	Rh		Cr	150
Th	10	Tb	0.36	Ru		V	540
Bi	140	Gd	0.89	Mo	23	Ti	0.51%
Pb	60	Eu	1.3	Nb	29	Sc	15
Tl	10	Sm	2.5	Zr	35	Ca	15%
Hg		Nd	45	Y	61	K	4.0%
Au		Pr	13	Sr	≅1,200	Cl	330
Pt		Ce	180	Rb	460	S	≅3,800
Ir		La	37	Br	14	P	0.61%
Os		Ba	480	Se	42	Si	40%
Re	*	Cs	6.0	As	890	Al	3.0%
W	17	I	11	Ge	6.3	Mg	10%
Ta	0.54	Te	2.7	Ga	60	Na	20%
Hf	0.99	Sb	10	Zn	360	F	9.0%
Lu	0.32	Sn	36	Cu	210	B	100
Yb	1.1	In	*	Ni	88	Be	2.3
Tm	0.12	Cd	13	Co	3.4	Li	390
Er	0.50	Ag	0.57	Fe	6.0%		
Ho	0.27	Pd		Mn	≅1,600		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 13. SPARK SOURCE MASS SPECTROSCOPY
CERAMICS PLANT
3μ-10μ PARTICULATES
(ppm)

U	5.6	Dy	4.5	Rh		Cr	120
Th	11	Tb	0.53	Ru		V	210
Bi	79	Gd	0.99	Mo	12	Ti	0.67%
Pb	33	Eu	1.8	Nb	32	Sc	40
Tl	5.3	Sm	3.6	Zr	38	Ca	15%
Hg		Nd	49	Y	38	K	4.0%
Au		Pr	9.9	Sr	≅1,400	Cl	200
Pt		Ce	200	Rb	370	S	≅4,700
Ir		La	27	Br	7.6	P	0.67%
Os		Ba	290	Se	20	Si	30%
Re	*	Cs	6.6	As	110	Al	3.0%
W	3.7	I	5.9	Ge	1.4	Mg	6%
Ta	0.60	Te	1.3	Ga	36	Na	15%
Hf	1.3	Sb	5.3	Zn	220	F	1.0%
Lu	0.36	Sn	9.2	Cu	93	B	230
Yb	1.8	In	*	Ni	36	Be	1.2
Tm	0.14	Cd	6.6	Co	18	Li	430
Er	1.0	Ag	0.64	Fe	6.0%		
Ho	0.30	Pd		Mn	≅1,300		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 14. SPARK SOURCE MASS SPECTROSCOPY
SLUDGE INCINERATOR
1μ-3μ PARTICULATES
(ppm)

U	11	Dy	1.8	Rh		Cr	≅4,000
Th	2.3	Tb	0.21	Ru		V	94
Bi	≅1,500	Gd	1.5	Mo	110	Ti	0.53%
Pb	≅1,300	Eu	0.97	Nb	30	Sc	3.8
Tl	2.5	Sm	1.1	Zr	210	Ca	>1%
Hg		Nd	47	Y	18	K	1.0%
Au	84	Pr	9.4	Sr	≅1,300	Cl	≅4,600
Pt		Ce	190	Rb	22	S	0.8%
Ir		La	39	Br	720	P	10.0%
Os		Ba	≅1,700	Se	73	Si	10%
Re	*	Cs	18	As	100	Al	0.9%
W	35	I	280	Ge	2.8	Mg	6.0%
Ta	1.4	Te	0.29	Ga	130	Na	10%
Hf	2.7	Sb	2.5	Zn	0.56%	F	≅4,300
Lu	0.72	Sn	1.0%	Cu	3.0%	B	82
Yb	0.29	In	*	Ni	≅4,500	Be	2.4
Tm	0.10	Cd	630	Co	850	Li	50
Er	0.26	Ag	600	Fe	7.0%		
Ho	0.13	Pd		Mn	850		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 15. SPARK SOURCE MASS SPECTROSCOPY
SLUDGE INCINERATOR
3μ-10μ PARTICULATES
(ppm)

U	31	Dy	2.5	Rh		Cr	2,300
Th	3.1	Tb	0.30	Ru		V	55
Bi	≅1,900	Gd	0.55	Mo	33	Ti	≅3,800
Pb	≅2,200	Eu	1.3	Nb	31	Sc	4.4
Tl	5.3	Sm	2.6	Zr	120	Ca	20%
Hg		Nd	55	Y	21	K	1.0%
Au	120	Pr	11	Sr	≅1,500	Cl	≅2,700
Pt		Ce	220	Rb	24	S	0.57%
Ir		La	42	Br	420	P	10.0%
Os		Ba	≅2,000	Se	52	Si	8%
Re	*	Cs	11	As	43	Al	0.69%
W	44	I	330	Ge	1.7	Mg	7.0%
Ta	3.4	Te	0.34	Ga	74	Na	8%
Hf	3.7	Sb	30	Zn	0.56%	F	≅2,500
Lu	0.37	Sn	0.5%	Cu	3.0%	B	65
Yb	0.37	In	*	Ni	≅3,000	Be	14
Tm	0.08	Cd	790	Co	≅1,000	Li	85
Er	0.31	Ag	500	Fe	8.0%		
Ho	0.13	Pd	16	Mn	≅1,000		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 16. SPARK SOURCE MASS SPECTROSCOPY†
STEEL PLANT COKE OVEN HEATER
1μ-3μ PARTICULATES
(ppm)

U	3.4 (16)	Dy		Rh		Cr	94 (170)
Th	0.24	Tb		Ru		V	38 (15.5)
Bi	48 (135)	Gd		Mo	17 (12)	Ti	8.0 (125)
Pb	940 (1,500)	Eu		Nb		Sc	>1%
Tl	7.5 (84)	Sm		Zr	0.66	Ca	≅1,100 (220)
Hg		Nd		Y	0.08	K	≅1,700 (1,500)
Au		Pr		Sr	1.2 (6.5)	Cl	17
Pt		Ce	0.10	Rb	21 (340)	S	>1% (7,250)
Ir		La	0.03	Br	1.1	P	41 (82)
Os		Ba	1.0 (15)	Se	9.8	Si	200 (2,500)
Re	*	Cs	0.53	As	3.9 (200)	Al	5.3 (17)
W	0.87	I	1.8 (5.8)	Ge	0.98	Mg	170
Ta	0.18	Te	0.85	Ga	4.0	Na	≅2,200 (700)
Hf		Sb	3.7	Zn	≅1,100 (850)	F	220 (100)
Lu		Sn	66 (81)	Cu	170 (190)	B	3.3 (15)
Yb		In	*	Ni	51	Be	0.25
Tm		Cd	9.4 (131)	Co	2.6 (11)	Li	6.1
Er		Ag	4.2 (35)	Fe	>1% (4,500)		
Ho		Pd		Mn	250 (82)		

*Internal standard.

†Data in parentheses generated by EMSL/RTP.

No data indicates "below detection limit of 0.1 ppmw."

TABLE 17. SPARK SOURCE MASS SPECTROSCOPY
OIL-FIRED POWER PLANT
1μ-3μ PARTICULATES
(ppm)

U	1.3	Dy	0.31	Rh		Cr	≅1,000
Th	1.3	Tb	0.04	Ru		V	2.0%
Bi	1.1	Gd	0.10	Mo	814	Ti	210
Pb	450	Eu	0.33	Nb	0.22	Sc	<0.08
Tl	0.78	Sm		Zr	2.2	Ca	1.0%
Hg		Nd	0.68	Y	13	K	≅3,500
Au		Pr	0.14	Sr	57	Cl	33
Pt		Ce	14	Rb	29	S	7.0%
Ir		La	0.52	Br	10	P	≅1,600
Os		Ba	280	Se	63	Si	≅4,500
Re	*	Cs	4.6	As	9.3	Al	3.40
W	2.4	I	0.61	Ge	4.7	Mg	0.59%
Ta	0.41	Te	0.12	Ga	190	Na	20.0%
Hf		Sb	7.8	Zn	900	F	31
Lu		Sn	14	Cu	300	B	16
Yb		In	*	Ni	5.2	Be	8.1
Tm		Cd	0.97	Co	220	Li	13
Er		Ag	4.4	Fe	1.0%		
Ho		Pd		Mn	220		

*Internal standard.

No data indicates "below detection limit of 0.1 ppmw."

STUDY NUMBER 16

DATA SOURCE:

LEVEL 1 ANALYSIS OF BITUMEN, STEAM CONDENSATE AND AN ARTIFICIAL MIXTURE

EPA Contract No. 68-02-1409,
Tasks 39 and 41

DATA STATUS:

Letter, January 1976

TO:

Mr. Larry D. Johnson
Control Systems Laboratory
Industrial Energy Research Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, North Carolina 27711

FROM

Peter W. Jones
Battelle-Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

GENERAL

Five samples (bitumen, steam condensate, an artificial mixture, an oil combustion effluent before addition of water to the oil, and an oil combustion effluent after addition of water to the oil) were subjected to Level 1 analysis. The oil combustion samples were composites of extracts of an adsorbent sampler, a filter, and solvent rinses of the probe and filter holder. Samples were analyzed according to the Technical Manual for Analysis of Organic Materials in Process Streams. The oil combustion samples were also analyzed for POM's by GC-MS. Copies of all of the actual spectra were attached to the letter.

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TABLE 1. LC FRACTIONATION

Fraction	Bitumen				Steam Condensate			
	TCO	Grav g	Total	Total	TCO	Grav g	Total	Total
LC 1		0.0089				.00004		
LC 2		.00089				.00002		
LC 3		.00081				.00003		
LC 4		.00035				.0018		
LC 5		.00028				.012		
LC 6		.0014				.00004		
LC 7		.00021				.00014		
LC 8		.00045				.00077		

Fraction	Artificial Mixture			
	TCO	Grav g	Total	Total
LC 1		.0043		
LC 2		.0015		
LC 3		.0012		
LC 4		.0036		
LC 5		.0071		
LC 6		.003		
LC 7		.014		
LC 8		.00081		

TABLE 2. LC FRACTIONATION
COMBINED EXTRACTS FROM AN OIL COMBUSTION EFFLUENT

Fraction	Before addition of water to oil				After addition of water to oil			
	TCO	Grav g	Total	Total	TCO	Grav g	Total	Total
LC 1		.0014				.006		
LC 2		.00003				.00031		
LC 3		.00019				.00026		
LC 4		.0033				.0029		
LC 5		.000077				.00026		
LC 6		.00038				.0015		
LC 7		.00022				.00019		
LC 8		.00045				.000039		

TABLE 3. IR REPORT
SAMPLE: BITUMEN

LC FRACTION	Wave number (cm^{-1})	Intensity	Assignments	Comments
1	1,690; 1,600	Strong	Aliphatic hydrocarbons Asphaltic materials	The 1690 band is due to a gross mixture of different carbonyl compounds. The 1600 band is due to structures such as highly condensed aromatics and quinones.
2			Same as fraction 1 plus aromatic materials	Possible POM's
3			Same as fraction 2	
4			Same as fraction 1	
5			Same as fraction 1 plus considerable -OH	Probably phenolic
6	1,025		Same as fraction 1 plus possible $Si0_2$*	$Si0_2$(1025) band weakest in fraction 6 and strongest in fraction 8
7	1,025		Same as fraction 1 plus possible $Si0_2$*	$Si0_2$(1025) band weakest in fraction 6 and strongest in fraction 8
8	1,025		Same as fraction 1 plus possible $Si0_2$*	$Si0_2$(1025) band weakest in fraction 6 and strongest in fraction 8

*$Si0_2$ is a probable assignment in view of the slight solubility of silica gel in methyl alcohol.

TABLE 4. IR REPORT
SAMPLE: STEAM CONDENSATE

LC FRACTION	Wave number (cm^{-1})	Intensity	Assignments (Comments)
1	---	Very weak	Aliphatic hydrocarbon
2	--- 1,725	Very weak Very weak	Aliphatic hydrocarbon Ester
3			Same as fraction 2
4			Same as fraction 2
5	1,040; 1,065	Strong	Same as fraction 2 plus a strong -OH which is aromatic and probably monosubstituted. The -OH is probably phenolic, but could possibly be a mixture of phenol and alcohol in view of band at 1040 and 1065 cm^{-1} which could be attributable to an alcohol.
6			Same as fraction 2
7*	1,600; 1,690		Same as fraction 2 plus probable condensed ring asphaltic species.
8*	1,600; 1,690		Same as fraction 2 plus probable condensed ring asphaltic species.

*Probable condensed ring asphaltic species stronger in fraction 8 than in fraction 7.

TABLE 5. IR REPORT
SAMPLE: ARTIFICIAL MIXTURE

LC FRACTION	Wave number (cm^{-1})	Intensity	Assignments (Comments)
1			Anthracene
2			Anthracene; an aromatic compound which could be chlorinated.
3			Aliphatic ester; same aromatic compound as fraction 2.
4			Phenol; the same aliphatic ester as fraction 3; a ketone; possibly an ether.
5			Phenol, 1-octanol
6			Aromatic amide, alkyl benzene sulfonate (acid or salt)
7			The same sulfonate as fraction 6; dimethyl sulfoxide; an unknown compound masked by the sulfoxide
8			The same sulfonate as fraction 6

TABLE 6. IR REPORT
SAMPLE: COMBINED EXTRACTS FROM AN OIL COMBUSTION EFFLUENT

LC fraction	Before and after addition of water to oil*
1	Trace of silicone grease (presumably a contaminant)
2	Trace of silicone grease (presumably a contaminant); a carbonyl compound; an aliphatic hydrocarbon (probably an ester)
3	Carbonyl compound and an aliphatic hydrocarbon as in fraction 2; an unknown orange-colored material (possibly a highly conjugated aromatic aldehyde or a quinone, both having aliphatic substitution [this material is frequently observed during POM analyses]).
4	Same as fraction 3
5	Same as fraction 3 plus an aromatic species which is probably a phenol
6	Aliphatic acid
7	Very weak hydrocarbon bands; bands at 1600 cm^{-1}, which are probably water on dissolved silica gel
8	Same as fraction 7

*Spectra of before and after addition of water to oil were identical.

STUDY NUMBER 17

DATA SOURCE:	**STATUS OF OCEAN INCINERATION OF ORGANIC CHLORIDES ABOARD MATHIAS III AS OF 29 JUNE 1976**
DATA STATUS:	Preliminary Report, June 1976
AUTHOR:	H. J. Fisher
CONTRACTOR:	TRW, Inc. Environmental Engineering Division One Space Park Redondo Beach, California 90278 Contract No. 68-02-2165
APPROVED BY:	Birch J. Matthews Program Manager
PROJECT OFFICER:	Ronald A. Venezia Chemical Processes Branch Industrial Environmental Research Laboratory U.S. Environmental Protection Agency Research Triangle Park, NC 27711

GENERAL

The ship Matthias III is an ocean-going incinerator designed to burn liquid and solid wastes. In a cooperative study involving EPA, the French Atomic Energy Commission, Meneba (a Dutch holding company) and its subsidiary, Stahl-und Blech-Bau, nonbiodegradable organic chloride compounds were incinerated aboard the Matthias III. The purpose of this study was threefold: to perform an initial evaluation of the capabilities of the Matthias III for burning organic chloride wastes; to become acquainted with the French analytical team; and to visit the French CEA and CERBOM laboratories.

The nonbiodegradable organic chloride compounds, when incinerated under ideal conditions, produce H_2O, N_2, CO_2, and HCl. The ocean's bicarbonate buffering system and inherent high chloride level will theoretically accommodate these effluent gases. To study incinerator efficiency, the amount of emitted HCl was compared to the amount of HCl produced by catalytic oxidation of effluent gases.

In this study, liquid-air burners were used with combustion temperature held at 1200°-1500° C. Two separate tanks (#8 and #6) of organic chloride wastes were burned. Tank #8 was described as a two-phase, complex mixture containing 5% Cl in the top layer and 36% Cl in the bottom layer. Tank #6 was more homogenous and contained 27% Cl. Some preliminary data from the burns of the two tanks are reported.

Some problems encountered in this study were: the 3-meter probe was too short for efficient sampling under some wind conditions; the flame was, at times, not contained in the stack; and on occasion, a black plume indicated incomplete combustion (determined by testing to be 98% complete). No Level 1 data are formatted from this preliminary report, but this summarization is included for future reference.

GASEOUS GRAB

A Level 1 gaseous grab sample was not obtained. Sampling of effluent gases was accomplished through a specially insulated and cooled Teflon-lined probe, connected by Teflon tubing to analytical apparati. HCl concentration and concurrent incinerator efficiency were tested using this sampling configuration, plus a microcoulometry cell. A developmental emission spectrophotometer was used for remote measurement of CO_2 and SO_2, but these results were not available at the time of the report.

SASS

A SASS train was not used in this study. Organic vapors from the sampling probe described under gaseous grab sampling were collected on

Tenax-GC cartridges for subsequent GC-MS analysis. Preliminary interpretation of analyses showed tank #8 effluent gases contained carbonic acid, chloride, benzene, styrenes, homologs of styrene, acetophenone, and a broad range of alkyl benzenes. Likewise, tank #6 contained ethyl chloride, trichlorofluoromethane, acetone, dichloromethane, octanol, trichloroethylene, and tetrachloroethylene. Tank #6 also showed some trace metals as indicated in Table 1 from the report.

TABLE 1. TRACE METALS IDENTIFIED IN EFFLUENT GASES FROM MATTHIAS III WHILE BURNING FROM TANK NUMBER 6
(Cadarache, preliminary data)

Cd	2.5 ppm*
Cr	0.5 ppm
Cu	18 ppm
Fe	140 ppm
Hg	1.2 ppm
Mn	7.5 ppm
Ni	6.5 ppm
Zn	62 ppm
Pb	18 ppm

*Chloride basis, e.g., Cd/Cl = $2.5/10^6$

FUGITIVE EMISSIONS

No sampling was performed in this study.

LIQUIDS AND SLURRIES

No sampling was performed in this study.

SOLIDS

No sampling was performed in this study.

STUDY NUMBER 18

DATA SOURCE:

EVALUATION OF THE SASS TRAIN AND LEVEL 1 SAMPLING AND ANALYSIS PROCEDURES MANUAL

DATA STATUS: Draft Report, November 1976

AUTHORS: C. A. Zee, D. G. Ackerman
J. F. Clausen, M. L. Kraft, S. L. Reynolds

CONTRACTOR: TRW Defense and Space Systems Group
One Space Park
Redondo Beach, California 90278
Contract No. 68-02-2165, Task 5

PROJECT OFFICER: Robert M. Statnick
Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

GENERAL

"The task objective was to test and evaluate the Source Assessment Sampling System (SASS) and the Level I Sampling and Analysis Procedures." Testing was done on combustion of #6 residual oil, doped with Hg, As, Se, and Sb, in a TRW Low-NO_x burner system. Combustion conditions were: flame temperature - 810° to 870° C; oil feed rate - 63.5 g/s; air feed rate - 103 kg/s. Two test runs were made. Approximate particulate loadings were: test I - 8.223 mg/dscm, and test II - 13.903 mg/dscm.

Specific recommendations were made for improving the SASS train and sampling procedures. For the SASS train, these included: (1) replacing Teflon O-rings with gaskets, (2) designing the XAD-2 canisters to have screwoff lids, (3) recalibrating the thermocouple on the small filter oven, (4) examining the welding materials to eliminate rust, (5) insulating the impinger box, and (6) using smaller corrosion-resistant packing containers for the XAD-2 canisters.

For sampling and analysis procedures, recommendations included: (1) specifying 30 m^3 as the dry standard volume for an SASS sample run; (2) extracting CH_2Cl_2 from a portion of the aqueous condensate rather than from the total condensate; (3) defining the weight of sample necessary to perform LC fractionation as 15 mg for a 30-m^3 sample; (4) cleaning particulate filters by aqua regia digestion rather than by Parr bombing; (5) expanding details on the Hg, As, and Sb procedures; (6) defining "dry weight" in the gravimetric procedures; and (7) using a small Soxhlet for extractions with several rechargings per sample. Problems also were noted with possible silica gel artifacts in LC fractions and incomplete Parr bomb combustion of the XAD-2 resin.

Conclusions of the study were that: (1) no organics were detected downstream of the XAD-2 module, (2) the majority or inorganics were collected on the particulate filter, (3) the APS/$AgNO_3$ solution was effective in scrubbing Hg, (4) the amount of an inorganic constituent could not be related directly to its level in the oil or doping solution, (5) some Level I analytical procedures needed more detail, and (6) Level I sampling procedures were "specific, easy-to-follow, and technically sound."

GASEOUS GRAB

No sampling was performed in this study.

SASS

The SASS train was modified as follows: No cyclones were used for particle sizing; all particulates were collected on filters which were pre-

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pared for SSMS by aqua regia digestion rather than by Parr bombing. A water- and air-cooled probe was substituted for the standard SASS probe. The XAD-2 resin was not removed from the resin module in the field and the CH_2Cl_2 extraction of the aqueous condensate was not performed in the field. Aqueous impinger catches were not combined (so that the distribution of As, Se, Hg, and Sb throughout the system could be analyzed). Sb analysis was done by AA rather than the specified Rhodamine B method.

FUGITIVE EMISSIONS

No sampling was performed in this study.

LIQUIDS AND SLURRIES

Sampling methods were not specified. The #6 residual fuel oil was tested by SSMS.

SOLIDS

No sampling was performed in this study.

TABLE 1. SPARK SOURCE MASS SPECTROSCOPY
XAD-2 RESIN--TEST 2
(μg/l of gas sampled)

First duplicate analysis

U	Dy	Rh	Cr *
Th	Tb	Ru	V *
Bi	Gd	Mo ≤0.008	Ti *
Pb 0.003	Eu	Nb	Sc <0.001
Tl	Sm	Zr 0.008	Ca 0.08
Hg	Nd	Y ≤0.003	K 0.1
Au	Pr	Sr 0.0005	Cl 0.06
Pt †	Ce ≤0.006	Rb <0.0008	S 0.08
Ir	La ≤0.003	Br ≤0.0005	P 0.03
Os	Ba 0.002	Se	Si *
Re	Cs *	As ≤0.001	Al *
W	I 0.08	Ge	Mg 0.11
Ta	Te	Ga <0.001	Na *
Hf	Sb	Zn 0.01	F ≅0.003
Lu	Sn	Cu *	B 0.003
Yb	In	Ni *	Be
Tm	Cd	Co ≤0.0008	Li <0.003
Er	Ag ≤0.002	Fe *	
Ho	Pd	Mn *	

Second duplicate analysis

U	Dy	Rh	Cr *
Th	Tb	Ru	V 0.0003
Bi	Gd	Mo <0.001	Ti *
Pb 0.1	Eu	Nb	Sc ≤0.001
Tl	Sm	Zr 0.017	Ca 0.08
Hg	Nd	Y ≤0.003	K *
Au	Pr	Sr 0.0005	Cl 0.04
Pt †	Ce <0.001	Rb ≤0.0008	S 0.14
Ir	La <0.001	Br ≤0.0005	P 0.06
Os	Ba 0.002	Se	Si 0.03
Re	Cs *	As ≤0.001	Al 0.006
W	I *	Ge	Mg 0.08
Ta	Te	Ga ≤0.001	Na *
Hf	Sb	Zn *	F ≅0.01
Lu	Sn	Cu *	B 0.04
Yb	In	Ni *	Be
Tm	Cd	Co ≤0.0008	Li <0.0003
Er	Ag ≤0.001	Fe *	
Ho	Pd	Mn 0.003	

* Blank value is greater than or equal to sample value.

†Platinum values do not necessarily relate to the source since Pt wire was used in the Parr bomb preparation of the resin sample.

NOTE: No data indicates "not detected"; minimum detectable limit is 0.0014 μg/l.

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY COMBINED IMPINGER SOLUTIONS (μg/l of gas sampled)

Test 1

U		Dy		Rh		Cr	0.02
Th		Tb		Ru		V	≤0.0006
Bi		Gd		Mo	≤0.005	Ti	≤0.003
Pb	≤0.004	Eu		Nb		Sc	≤0.0005
Tl		Sm		Zr	≤0.004	Ca	0.06
Hg		Nd		Y		K	0.2
Au		Pr		Sr	<0.0007	Cl	0.07
Pt	<0.0007	Ce		Rb		S	0.9
Ir		La		Br	≤0.0004	P	*
Os		Ba	0.0007	Se		Si	0.01
Re		Cs	≤0.0005	As	<0.0007	Al	0.006
W		I	≤0.0005	Ge		Mg	0.03
Ta		Te		Ga	≤0.0006	Na	0.2
Hf		Sb		Zn	≤0.009	F	≅0.004
Lu		Sn	≤0.004	Cu	0.001	B	≤0.005
Yb		In		Ni	0.01	Be	
Tm		Cd		Co	≤0.001	Li	≤0.0001
Er		Ag		Fe	0.1		
Ho		Pd		Mn	0.001		

Test 2

U		Dy		Rh		Cr	0.02
Th		Tb		Ru		V	<0.001
Bi		Gd		Mo	<0.001	Ti	≤0.01
Pb	0.007	Eu		Nb		Sc	≤0.001
Tl		Sm		Zr	≤0.4	Ca	0.04
Hg		Nd		Y		K	0.01
Au		Pr		Sr	≤0.001	Cl	0.03
Pt	≤0.004	Ce		Rb		S	0.01
Ir		La		Br	≤0.001	P	*
Os		Ba	<0.001	Se		Si	0.03
Re		Cs	<0.001	As	≤0.0008	Al	0.005
W		I	≤0.0009	Ge		Mg	0.04
Ta		Te		Ga	<0.001	Na	0.03
Hf		Sb		Zn	≤0.003	F	≅0.1
Lu		Sn	<0.001	Cu	*	B	≤0.001
Yb		In		Ni	0.009	Be	
Tm		Cd		Co	<0.001	Li	≤0.0007
Er		Ag		Fe	0.1		
Ho		Pd		Mn	0.002		

*Blank value is greater than or equal to sample value.

NOTE: No data indicates "not detected;" minimum detectable limit is 0.007 μg/l for test 1 and 0.0013 μg/l for test 2.

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY
SASS FILTER--AQUA REGIA DIGEST*
(μg/l of sample gas)

Test 1

U		Dy		Rh		Cr	0.03
Th		Tb		Ru		V	0.05
Bi	≤0.0007	Gd		Mo		Ti	0.005
Pb	0.01	Eu		Nb		Sc	<0.0005
Tl	≤0.001	Sm		Zr	≤0.001	Ca	≥0.06
Hg		Nd		Y		K	0.007
Au		Pr		Sr	0.004	Cl	†
Pt		Ce		Rb	≤0.0003	S	0.07
Ir		La		Br	≤0.0001	P	0.02
Os		Ba	≤0.005	Se	≤0.0003	Si	0.03
Re		Cs	≤0.0003	As	0.01	Al	0.03
W	<0.001	I		Ge		Mg	>0.06
Ta	≤0.0003	Te		Ga	≤0.0004	Na	>0.03
Hf		Sb	<0.0003	Zn	0.0007	F	†
Lu		Sn		Cu	0.002	B	0.002
Yb		In		Ni	0.07	Be	<0.0003
Tm		Cd		Co	0.01	Li	0.0001
Er		Ag		Fe	0.07		
Ho		Pd		Mn	0.002		

Test 2

U		Dy		Rh		Cr	0.01
Th		Tb		Ru		V	0.04
Bi	<0.0007	Gd		Mo		Ti	†
Pb	0.007	Eu		Nb		Sc	≤0.0003
Tl	≤0.004	Sm		Zr	<0.0007	Ca	0.1
Hg		Nd		Y		K	†
Au		Pr		Sr	0.0001	Cl	†
Pt		Ce		Rb	0.003	S	>0.1
Ir		La		Br	≤0.0003	P	†
Os		Ba	≤0.003	Se	≤0.002	Si	0.03
Re		Cs	≤0.02	As	0.03	Al	0.09
W	<0.0007	I		Ge		Mg	0.07
Ta	<0.0007	Te		Ga	≤0.001	Na	>0.1
Hf		Sb	≤0.004	Zn	0.002	F	†
Lu		Sn		Cu	0.0006	B	0.01
Yb		In		Ni	0.03	Be	≤0.0001
Tm		Cd		Co	0.005	Li	†
Er		Ag		Fe	0.04		
Ho		Pd		Mn	0.001		

*No cyclones were used in this study.

†Blank value greater than or equal to sample value.

NOTE: No data indicates "not detected"; minimum detectable limits are 0.003 μg/l for test 1 and 0.007 μg/l for test 2.

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY
XAD-2 RESIN
(μg/l of gas sampled)

Test 1							
U		Dy		Rh		Cr	*
Th		Tb		Ru		V	0.005
Bi		Gd		Mo	≤0.01	Ti	*
Pb	0.04	Eu		Nb		Sc	<0.002
Tl		Sm		Zr	0.01	Ca	0.6
Hg		Nd		Y	<0.003	K	1.4
Au		Pr		Sr	0.001	Cl	0.06
Pt	†	Ce	≤0.008	Rb	≤0.002	S	0.08
Ir		La	≤0.005	Br	≤0.001	P	0.08
Os		Ba	0.02	Se		Si	*
Re		Cs	*	As	≤0.001	Al	0.005
W		I	0.005	Ge		Mg	0.08
Ta		Te		Ga	≤0.005	Na	>1
Hf		Sb		Zn	0.05	F	≅0.04
Lu		Sn		Cu	0.05	B	0.04
Yb		In		Ni	*	Be	
Tm		Cd		Co	≤0.002	Li	<0.0003
Er		Ag	≤0.003	Fe	0.03		
Ho		Pd		Mn	*		

*Blank value greater than or equal to sample value.

†Platinum values do not necessarily relate to the source since Pt wire was used by the Parr bomb preparation of the resin samples.

NOTE: No data indicates "not detected"; minimum detectable limit is 0.0024 μg/l.

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY NUMBER 6 RESIDUAL OIL (μg/g)

U		Dy		Rh		Cr	70
Th		Tb		Ru		V	10
Bi		Gd		Mo		Ti	8
Pb		Eu		Nb		Sc	≤2
Tl		Sm		Zr	6	Ca	40
Hg		Nd		Y		K	20
Au		Pr		Sr	3	Cl	40
Pt		Ce		Rb		S	200
Ir		La		Br	≤0.8	P	40
Os		Ba	2	Se		Si	20
Re		Cs		As	2	Al	5
W	≤9	I	1	Ge		Mg	40
Ta	7	Te		Ga		Na	100
Hf		Sb		Zn	7	F	≅2
Lu		Sn		Cu	20	B	1
Yb		In		Ni	300	Be	
Tm		Cd		Co	2	Li	<0.1
Er		Ag	2	Fe	80		
Ho		Pd		Mn	20		

NOTE: No data indicates "not detected."

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY BLANK VALUES FOR AQUA REGIA DIGESTS* (Total μg)

U		Dy		Rh		Cr	20
Th		Tb		Ru		V	4
Bi	<12	Gd		Mo		Ti	60
Pb	60	Eu		Nb		Sc	≤6
Tl	<12	Sm		Zr	<12	Ca	400
Hg		Nd		Y		K	200
Au		Pr		Sr	10	Cl	180
Pt		Ce		Rb	60	S	20
Ir		La		Br	<12	P	600
Os		Ba	<12	Se	<12	Si	400
Re		Cs	<12	As	8	Al	200
W	<12	I		Ge		Mg	200
Ta	18	Te		Ga	<12	Na	1,200
Hf		Sb	<12	Zn	20	F	≅120
Lu		Sn		Cu	10	B	20
Yb		In		Ni	40	Be	<8
Tm		Cd		Co	6	Li	2
Er		Ag		Fe	200		
Ho		Pd		Mn	4		

*One filter used.

NOTE: No data indicates "not detected."

TABLE 7. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS
SASS TRAIN SAMPLES
(μg)

Sample	Test 1		
	Hg	Sb	As*
Filter	0.16	<30	1,080
Filter blank	0.20	<30	1
XAD-2 resin	14.35	3,590	<115
XAD-2 blank	6.1	4,400	<115
Condensate	1.2	<146	1
1st impinger	1.9	708	209
2nd impinger	2.7	<364	74
3rd impinger	1.3	<80	<1
TOTAL	15.35	708	1,363

	Test 2		
Filter	0.88 (1.44)†	908 (1,572)†	7,360 (7,520)†
Filter blank	0.40	<60	2
XAD-2 resin	62.56 (49.59)†	4,470 (4,230)†	5,960 (5,834)†
XAD-2 blank	6.2	4,400	<120
Condensate	15.5	<396	1,954
1st impinger	24.0	235	388
2nd impinger	1,515	84	<1
3rd impinger	371	<65	<1
TOTAL	1,976	1,559	15,677

*As by colorimetric/SDDC method.
†Duplicate analysis.

From p. 33 of the document:

"The analyses for mercury, arsenic, antimony and selenium were performed on each component sample from the SASS without making any combinations as called for in the Level I manual. In addition, nitric acid rinses were made of the quartz probe liner and the heat traced Teflon sample line and were also analyzed. There was a factor of 4 to 5 difference in the feed rate of the doping solution between Tests I and II."

TABLE 8. GAS CHROMATOGRAPHY FOR C_7-C_{17} SASS TRAIN SAMPLES

Sample	Range	Volatile weight, mg/m^3*	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Probe rinse Test 1	GC7 90 - 110	24.1	N.R.	Probe rinse + filter extract 222.6	
	GC8 110 - 140	(0.02)			
	GC9 140 - 160	(0.02)			
	GC10 160 - 180	0.02			
	GC11 180 - 200	(0.02)			
	GC12 200 - 220	(0.02)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
Probe rinse Test 2	GC7 90 - 110	(0.02)	N.R.	Probe rinse + filter extract 314.8	
	GC8 110 - 140	(0.02)			
	GC9 140 - 160	(0.02)			
	GC10 160 - 180	(0.02)			
	GC11 180 - 200	(0.02)			
	GC12 200 - 220	(0.02)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
Filter extract Test 1	GC7 90 - 110	(0.01)	N.R.	Combined with probe rinse for grav.	
	GC8 110 - 140	(0.01)			
	GC9 140 - 160	(0.01)			
	GC10 160 - 180	(0.01)			
	GC11 180 - 200	(0.01)			
	GC12 200 - 220	(0.01)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				

*Values in parentheses are lower detectable limits for samples for which no peaks were found.

TABLE 8 (con.)

Sample	Range	Volatile weight, mg/m^3*	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Filter extract Test 2	GC7 90 - 110	(0.01)	N.R.	Combined with probe rinse for grav.	
	GC8 110 - 140	(0.01)			
	GC9 140 - 160	(0.01)			
	GC10 160 - 180	(0.01)			
	GC11 180 - 200	(0.01)			
	GC12 200 - 220	(0.01)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
Module rinse Test 1	GC7 90 - 110	(0.02)	N.R.	Combined with resin extract and condensate extract for grav.	
	GC8 110 - 140	(0.02)			
	GC9 140 - 160	0.027			
	GC10 160 - 180	(0.02)			
	GC11 180 - 200	(0.02)			
	GC12 200 - 220	(0.02)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
Module rinse Test 2	GC7 90 - 110	(0.02)	N.R.	Combined with resin extract and condensate extract for grav.	
	GC8 110 - 140	0.038			
	GC9 140 - 160	(0.02)			
	GC10 160 - 180	(0.02)			
	GC11 180 - 200	(0.02)			
	GC12 200 - 220	(0.02)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				

*Values in parentheses are lower detectable limits for samples for which no peaks were found.

TABLE 8 (con.)

Sample	Range	Volatile weight, mg/m^3*	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Resin extract Test 1	GC7 90 - 110	(0.01)	N.R.	Module rinse + resin extract + condensate extract 49.8	
	GC8 110 - 140	0.98			
	GC9 140 - 160	0.039			
	GC10 160 - 180	0.72			
	GC11 180 - 200	1.7			
	GC12 200 - 220	1.1			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
Resin extract Test 2	GC7 90 - 110	(0.01)	N.R.	Module rinse + resin extract + condensate extract 38.7	
	GC8 110 - 140	0.35			
	GC9 140 - 160	0.038			
	GC10 160 - 180	0.073			
	GC11 180 - 200	3.4			
	GC12 200 - 220	2.0			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
Condensate extract Test 1	GC7 90 - 110	(0.005)	N.R.	Combined with resin extract and module rinse	
	GC8 110 - 140	(0.005)			
	GC9 140 - 160	(0.005)			
	GC10 160 - 180	(0.005)			
	GC11 180 - 200	(0.005)			
	GC12 200 - 220	(0.005)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				

*Values in parentheses are lower detectable limits for samples for which no peaks were found.

TABLE 8 (con.)

Sample	Range	Volatile weight, mg/m^3*	No. of peaks	Gravimetric nonvolatile weight, mg	Total organic, mg
Condensate extract Test 2	GC7 90 - 110	(0.007)	N.R.	Combined with resin extract and module rinse	
	GC8 110 - 140	(0.007)			
	GC9 140 - 160	(0.007)			
	GC10 160 - 180	(0.007)			
	GC11 180 - 200	(0.007)			
	GC12 200 - 220	(0.007)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
Heat traced sample line rinse (for both runs)	GC7 90 - 110	(0.01)	N.R.	0.082	
	GC8 110 - 140	13.8			
	GC9 140 - 160	(0.01)			
	GC10 160 - 180	(0.01)			
	GC11 180 - 200	(0.01)			
	GC12 200 - 220	(0.01)			
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				
	GC7 90 - 110				
	GC8 110 - 140				
	GC9 140 - 160				
	GC10 160 - 180				
	GC11 180 - 200				
	GC12 200 - 220				
	GC13				
	GC14				
	GC15				
	GC16				
	GC17				

*Values in parentheses are lower detectable limits for samples for which no peaks were found.

TABLE 9. LC FRACTIONATION

	Run 1 Probe rinse and filter extract				Run 2 Probe rinse and filter extract			
Fraction	TCO mg*	Grav mg	Total mg	Total† mg/m^3	TCO mg*	Grav mg	Total mg	Total† mg/m^3
LC 1		0.565		0.020		0.0		0
LC 2		0.0		0		0.0		0
LC 3		0.0		0		0.0		0
LC 4		0.0		0		0.0		0
LC 5		0.0		0		0.0		0
LC 6		0.521		0.019		0.347		0.013
LC 7		36.444		1.32		57.753		2.12
LC 8		29.441		1.06		23.937		0.880

	Run 1 Module rinse + resin extract + condensate extract				Run 2 Module rinse + resin extract + condensate extract			
Fraction	TCO mg*	Grav mg	Total mg	Total† mg/m^3	TCO mg*	Grav mg	Total mg	Total† mg/m^3
LC 1		0.418		0.015		1.270		0.047
LC 2		0.0		0		0.218		0.008
LC 3		0.343		0.012		0.544		0.020
LC 4		0.409		0.015		0.533		0.020
LC 5		0.245		0.009		0.177		0.007
LC 6		2.080		0.075		2.029		0.075
LC 7		2.429		0.088		2.467		0.091
LC 8		12.746		0.460		10.064		0.370

*TCO was not performed in this study.

†Total volumes sampled: Run 1 - 27.7 m^3, Run 2 - 27.2 m^3.

TABLE 10. IR REPORT: CONCENTRATED SAMPLES BEFORE LC

Run	Findings
1	Probe Rinse + Filter Extract contains alkanes, esters, ketones.
2	Probe Rinse + Filter Extract contains water, silicone, alkanes, ethers, sulfonic acids.
1	Module rinse + resin extract + condensate extract contains alkanes, alkenes, aromatic esters, amides, low substituted aromatics, ethers, ketones.
2	Module rinse + resin extract + condensate extract contains aliphatic ethers, water, alcohols.
Blank	Module rinse + resin extract + condensate extract contains alkanes, ketones, aromatic esters. Heat traced line rinse for both runs contains alkanes, alkenes, amides, aromatic esters.

TABLE 11. IR REPORT: SAMPLES AFTER LC FRACTIONATION

LC	Run 1 Probe rinse + filter extract	Run 2 Probe rinse + filter extract	Run 1 Module rinse + resin extract + condensate extract	Run 2 Module rinse + resin extract + condensate extract
1	Alkanes	*	Alkanes, trace silicones	Alkanes
2	*	*	*	Alkanes, trace alkenes, aromatics
3	*	*	Epoxides, low substituted aromatics, olefins, ethers, trace silicones	Silicones, ethers aromatics
4	*	*	Trace silicones, aromatic esters, phthalate esters	Esters, aromatics, ketones, ethers
5	*	*	Aromatic esters, phthalate esters	Ketones, esters aromatics, phthalate esters
6	Phthalate esters	Aromatic esters, phthalate esters	Phthalate esters	Aromatics, aromatic esters
7	Epoxides, ethers silicones, alcohols	Epoxides, sulfonic acids, silicones, water, alcohols	Water, alcohols	Phthalate esters alcohols
8	Epoxides, ethers silicones, alcohols, water	Ethers, silicones sulfonic acids, alcohols, water	Phenols, sulfonic acids	Aromatic esters, aromatics, alcohols, water

*Insufficient sample to analyze.

TABLE 12. LRMS REPORT
PROBE RINSE + FILTER EXTRACT

1. **Categories Present**

Run	LC	Intensity	Category	MW Range
1	7	Major	Water	N.R.
		Minor	Hydrocarbons to m/e 200	
		Major	HCl	
		Major	SO_2 alkyl sulfonates, alkyl sulfinates	
		Major	Alkyl pyrroles, substituted pyridines, or anilines	
		Trace	Indenes, substituted naphthyl compounds, and/or aliphatic esters	
		Minor	C_9H_{11}, substituted benzene compounds	
		Major	Five compounds all believed to be siloxanes	
		Minor	Phthalate esters	
		Trace	Substituted anthracene compounds	

2. **Subcategories, Specific Compounds**

Run	LC	Intensity	Compound	m/e	Composition
1	7	---	N.R.	---	---

3. **Other**

Intensity: N,R.

NOTE: LRMS analysis was run on LC fractions with dry weights greater than 15 $mg/30\ m^3$ of gas sampled.

TABLE 12 (con.)

1. Categories Present

Run	LC	Intensity	Category	MW Range
1	8	Major	HCL	N.R.
		Major	CH_3OH	
		Major	SO_2, alkyl sulfonates, alkyl sulfinates	
		Major	Alkyl pyrroles, substituted pyridenes, and anilines	
		Minor to Trace	3 compounds believed to be siloxanes	
		Major	Phthalates	

2. Subcategories, Specific Compounds

Run	LC	Intensity	Compound	m/e	Composition
1	8	---	N.R.	---	---

3. Other

"This sample is somewhat dissimilar from the other three samples because it appears to have fewer components and the resulting spectrum is less complex."

Intensity
N.R.

NOTE: LRMS analysis was run on LC fractions with dry weights greater than 15 mg/30 m^3 of gas sampled.

TABLE 12 (con.)

1. Categories Present

Run	LC	Intensity	Category	MW Range
2	7	Minor	Hydrocarbons to m/e >300	N.R.
		Major	HCl	
		Major	CH_3OH	
		Major	H_2O	
		Minor	H_2SO_4 tentative (can also be C_6H_{10}, furfural alkanoates or C_7H_{14}; substituted thiophenes)	
		Major	Sulfur oxides, sulfanates, sulfinates	
		Minor	Phthalate esters	
		Major	Siloxane compounds	

2. Subcategories, Specific Compounds

Run	LC	Intensity	Compound	m/e	Composition
2	7	---	N.R.	---	---

3. Other

Intensity
N.R.

NOTE: LRMS analysis was run on LC fractions with dry weights greater than 15 $mg/30\ m^3$ of gas sampled.

TABLE 12 (con.)

1. Categories Present

Run	LC	Intensity	Category	MW Range
2	8	Minor	Hydrocarbons, detected to mass 200	N.R.
		Major	HCl	
		Minor	CH_3Cl	
		Moderate	C_5H_6N, aklyl pyrroles, substituted pyridines or anilines	
		Trace	CH_2Cl_2	
		Moderate	C_7H_{11}, alkyl diene or cycloalkane fragments	
		Major	Silicone compound(s) $C_5H_{13}O_2Si$ or substituted benzene compounds	
		Minor	Methyl siloxanes	
		Minor	Dichlorobenzene (?)	
		Minor	$(CH_3)_3Si\text{-}O\text{-}Si(CH_3)_2$	
		Moderate	Phthalate esters	
		Minor	Trichlorobenzene	
		Trace	Substituted anthracene (?)	
		Minor	Methyl Siloxanes	
		Minor	Esters of adipic acid	
		Minor	$(CH_3)_7Si_4O_4$ (methylsiloxanes)	

2. Subcategories, Specific Compounds

Run	LC	Intensity	Compound	m/e	Composition
2	8	---	N.R.	---	---

3. Other

Intensity: N,R.

NOTE: LRMS analysis was run on LC fractions with dry weights greater than 15 mg/30 m^3 of gas sampled.

STUDY NUMBER 19

DATA SOURCE:

SOURCE ASSESSMENT:
TEXTILE PLANT WASTEWATER TOXICS STUDY

DATA STATUS: Preliminary Draft, December 1977

AUTHOR: G. D. Rawlings

CONTRACTOR:
Monsanto Research Corporation
1515 Nicholas Road
Dayton, Ohio 45407
Contract No. 68-02-1874
ROAP No. 21AXM-071
Program Element No. 1AB015

TASK OFFICER:
Max Samfield
Industrial Environmental Research Laboratory
Office of Energy, Minerals, and Industry
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

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GENERAL

This manual covers the period from January 1977 to December 1977 in which Monsanto Research Corporation (MRC) compiled data of an ongoing study of effluents from 23 textile plants. MRC, in using a two-phased approach, reports data from Phase I in which baseline toxicity is gathered on the 23 plants and ranks the plants in order of toxicity. (Phase 2 will be to determine degrees of toxicity removal of the best of seven possible tertiary systems as applied to 10 plants chosen from Phase I.)

This study coincides with a study by EPA/ATMI (Environmental Protection Agency/American Textile Manufacturers Institute) identified as BATEA (best available technology economically available), in which a two-phased approach is also used. Phase I is to determine the best available technology for removing criteria pollutants (BOD_5, COD, color, sulfide, pH, chromium, phenols, TSS), priority pollutants (129 compounds), and toxic materials (arsenic, mercury, etc.) with Phase II following to determine which technology is economically available. The objective of both studies is to determine BATEA for toxicity removal in textile wastewater.

Phase I of the EPA/MRC study was already under way when it was decided to include Level 1 Environmental Assessment procedures; therefore, only 15 plants were sampled and tested under this plan. Biological testing was performed on 23 plants.

IR showed presence of aliphatic HC, C=0 esters and acids, aromatics, phthalate esters, and fatty acid groups. LRMS identified paraffinic/olefinic alkyl benzenes, alcoholic esters, di-n-actyl phthalate, bis (hydroxy-t-butyl phenol) propane, tri-t-butyl benzene, alkyl phenols, dichloroaniline, toluene-sulfonyl groups, vinyl stearate, and azo compounds. Bioassay results of secondary effluent of 23 plants indicate 7 to be sufficiently toxic enough for Phase II study. Most toxic were plants N, A, L, T, C, P, and S; least toxic were V and W; plant R was also included due to improper sampling during Phase 1. Plants B, D, E, F, G, H, J, K, M, U, X, Y, and Z were characterized as nontoxic.

C_7-C_{12} hydrocarbons were less than 1.0 g/m^3 (ppm) in 13 of the 14 plants tested with Plant X containing 3.0 g/m^3 of the C_7-C_{12} hydrocarbons.

A deviation from Level 1 procedures was approved by EPA in which methylene chloride extractions were not performed in the field due to the emulsions formed with textile effluents. The emulsion is best broken down in the lab. In addition, the CH_2Cl_2 extract was not evaporated to dryness prior to LC/LRMS. Two additional steps are the use of GC/MS to quantitate C_7-C_{12} and the use of a solvent exchange to alter the polarity of the medium containing the organics.

GASEOUS GRAB

No sampling was performed in this study.

SASS

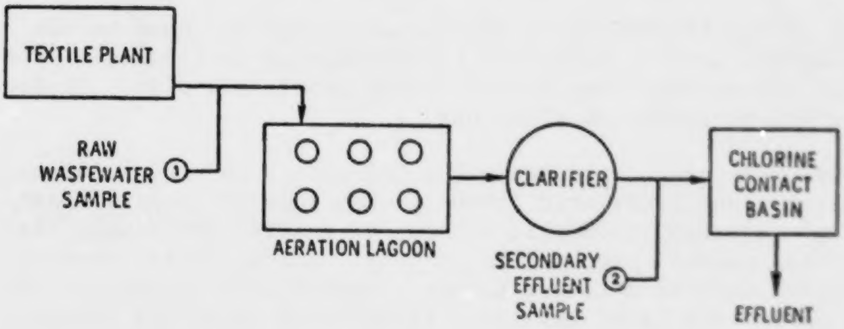
No sampling was performed in this study.

FUGITIVE EMISSIONS

No sampling was performed in this study.

LIQUIDS AND SLURRIES

Composite sampling of raw wastewater was performed at a location between the plant and aeration ponds. Secondary effluents were composite grab sampled over a period of 8 hours at a location between the clarifier and the chlorine contact basin. Plant R samples were taken between the aeration lagoon and settling basin and Plant Y samples were taken after the finishing pond. The effluents were filtered prior to CH_2Cl_2 extraction of the filtrate. DO, pH, color, odor, and cyanide samples were not extracted prior to analysis. The CH_2Cl_2 extract was analyzed for C_7-C_{12} organics. The extract was concentrated by K-D and analyzed by IR, LC Fractionation, and LRMS.


TEXTILE PLANT
RAW WASTEWATER SAMPLE ①
AERATION LAGOON
CLARIFIER
SECONDARY EFFLUENT SAMPLE ②
CHLORINE CONTACT BASIN
EFFLUENT

Phase I sampling locations.

SOLIDS

Suspended solids as removed from the wastewater were the only solids studied. Leachate analysis was not performed because each plant met EPA effluent standards for TSS. Filter paper was ashed by low temperature plasma, digested, and analyzed for trace elements by SSMS.

TABLE 1. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT A (μg/l)*

U	<0.04	Dy	<0.01	Rh		Cr	56
Th	<0.04	Tb	<0.03	Ru		V	318
Bi	3.0	Gd	<0.04	Mo	17	Ti	9.5
Pb	42	Eu	<0.04	Nb	0.03	Sc	0.07
Tl	<0.04	Sm	<0.07	Zr	1.9	Ca	1,220
Hg	NR	Nd	<0.09	Y	0.08	K	265
Au		Pr	<0.05	Sr	6.0	Cl	15
Pt		Ce	1.3	Rb	1.0	S	1,987
Ir		La	0.91	Br	3.0	P	1,590
Os		Ba	15	Se	0.25	Si	450
Re	†	Cs		As	3.4	Al	874
W	<0.08	I	0.33	Ge	0.03	Mg	702
Ta	<0.03	Te		Ga	0.13	Na	4,636
Hf	<0.11	Sb	1.6	Zn	300	F	28
Lu		Sn	13	Cu	172	B	NR
Yb	<0.04	In	†	Ni	26	Be	<0.04
Tm	<0.03	Cd	2.5	Co	2.0	Li	1.3
Er	<0.03	Ag	1.1	Ie	582		
Ho		Pd		Mn	11		

*Detection limit--0.01 μg/l
†Internal standard
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 2. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT B (μg/l)*

U		Dy		Rh		Cr	0.5
Th		Tb		Ru		V	0.02
Bi	0.05	Gd		Mo		Ti	0.3
Pb	0.2	Eu		Nb	0.04	Sc	
Tl		Sm		Zr		Ca	30
Hg	NR	Nd	0.1	Y		K	11
Au		Pr		Sr	0.1	Cl	0.3
Pt		Ce	0.02	Rb	0.08	S	24
Ir		La	0.01	Br	0.02	P	120
Os		Ba	1.8	Se		Si	340
Re	†	Cs		As		Al	25
W		I		Ge		Mg	130
Ta	0.003‡	Te		Ga		Na	240
Hf	<0.02	Sb		Zn	1.8	F	6.8
Lu		Sn	0.01	Cu	0.3	B	NR
Yb		In	†	Ni	0.07	Be	
Tm		Cd		Co		Li	0.04
Er		Ag		Ie	13		
Ho		Pd		Mn	0.3		

*Detection limit--0.01 μg/l
†Internal standard
‡Instrument source
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 3. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT C (μg/l)*

U		Dy		Rh		Cr	0.8
Th		Tb		Ru		V	1.1
Bi	0.7	Gd	0.07	Mo	0.1	Ti	0.8
Pb	0.6	Eu		Nb		Sc	0.1
Tl	0.07	Sm	0.07	Zr	0.2	Ca	83
Hg	NR	Nd		Y	0.03	K	330
Au		Pr		Sr	1.6	Cl	43
Pt		Ce	0.1	Rb	2.8	S	110
Ir		La	0.07	Br	1.1	P	700
Os		Ba	57	Se		Si	1,100
Re	†	Cs	0.03	As	0.07	Al	530
W	0.07	I	0.03	Ge		Mg	160
Ta	0.01‡	Te		Ga		Na	2,800
Hf	0.1	Sb	0.1	Zn	29	F	33
Lu		Sn	0.2	Cu	4	B	NR
Yb	0.07	In	†	Ni	0.9	Be	
Tm		Cd	0.07	Co	0.07	Li	0.3
Er	0.07	Ag		Ie	20		
Ho		Pd		Mn	0.9		

*Detection limit--0.05 μg/l

†Internal standard

‡Instrument source

NR = Not reported

No data indicates "not detected above detection limit."

TABLE 4. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT E (μg/l)*

U		Dy		Rh		Cr	0.07
Th		Tb		Ru		V	
Bi	0.07	Gd		Mo	0.04	Ti	0.05
Pb	0.09	Eu		Nb		Sc	<0.04
Tl		Sm		Zr	0.02	Ca	3.9
Hg	NR	Nd		Y		K	110
Au		Pr	0.02	Sr	0.32	Cl	18
Pt		Ce	0.02	Rb	0.49	S	0.74
Ir		La	0.02	Br	2.8	P	0.04
Os		Ba	0.09	Se		Si	3.5
Re	†	Cs		As	<0.07	Al	18
W	0.09	I	0.16	Ge		Mg	5.8
Ta		Te		Ga		Na	972
Hf	<0.05	Sb	0.02	Zn	0.12	F	11
Lu		Sn	0.04	Cu	0.04	B	NR
Yb	<0.04	In	†	Ni	0.05	Be	
Tm		Cd	0.02	Co		Li	0.02
Er		Ag		le	0.94		
Ho		Pd		Mn	0.05		

*Detection limit--<0.02 μg/l

†Internal standard

NR = Not reported

No data indicates "not detected above detection limit."

TABLE 5. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT F (µg/1)*

U		Dy		Rh		Cr	2.0
Th		Tb		Ru		V	0.1
Bi		Gd		Mo	0.1	Ti	1.4
Pb	4.0	Eu		Nb		Sc	<0.1
Tl		Sm		Zr	0.4	Ca	120
Hg	NR	Nd		Y		K	87
Au		Pr		Sr	0.6	Cl	34
Pt		Ce	0.1	Rb	0.1	S	106
Ir		La	0.05	Br	1.6	P	290
Os		Ba	4.3	Se		Si	820
Re	†	Cs		As	0.5	Al	42
W		I		Ge		Mg	30
Ta	0.002‡	Te		Ga		Na	1,100
Hf	<0.1	Sb	0.4	Zn	39	F	82
Lu		Sn	1.3	Cu	29	B	NR
Yb		In	†	Ni	1.2	Be	
Tm		Cd	0.2	Co	0.1	Li	NR
Er		Ag		Ie	67		
Ho		Pd		Mn	1.1		

*Detection limit--0.05 µg/1
†Internal standard
‡Instrument source
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 6. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT K (µg/1)*

U		Dy		Rh		Cr	0.8
Th		Tb		Ru		V	
Bi		Gd		Mo	0.06	Ti	0.09
Pb	0.51	Eu		Nb		Sc	<0.03
Tl	0.01	Sm		Zr	0.06	Ca	100
Hg	NR	Nd		Y		K	23
Au		Pr	0.01	Sr	0.06	Cl	1.2
Pt		Ce	0.03	Rb	0.3	S	18
Ir		La	0.04	Br	0.03	P	9.7
Os		Ba	0.84	Se		Si	23
Re	†	Cs		As	<0.04	Al	45
W	0.06	I	0.03	Ge		Mg	7.2
Ta		Te		Ga		Na	308
Hf	<0.03	Sb	0.01	Zn	0.75	F	1.4
Lu		Sn	0.04	Cu	0.97	B	NR
Yb		In	†	Ni	0.32	Be	
Tm		Cd		Co		Li	0.03
Er		Ag		Ie	3.2		
Ho		Pd		Mn	0.1		

*Detection limit--0.01 µg/1
†Internal standard
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 7. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT L (μg/l)*

U		Dy		Rh		Cr	13
Th		Tb		Ru		V	1.0
Bi	0.05	Gd		Mo	0.1	Ti	3.2
Pb	9	Eu		Nb		Sc	<0.1
Tl		Sm	0.3	Zr	0.3	Ca	1,200
Hg	NR	Nd	0.05	Y		K	95
Au		Pr	0.05	Sr	1.0	Cl	37
Pt		Ce	0.2	Rb	0.3	S	360
Ir		La	0.1	Br	0.4	P	1,200
Os		Ba	23	Se		Si	4,600
Re	†	Cs		As		Al	440
W		I		Ge		Mg	650
Ta	0.001‡	Te		Ga		Na	5,000
Hf	<0.1	Sb	0.2	Zn	1,900	F	33
Lu		Sn	0.2	Cu	31	B	NR
Yb		In	†	Ni	3.6	Be	
Tm		Cd	0.05	Co	0.05	Li	0.3
Er		Ag		Ie	380		
Ho		Pd		Mn	2.2		

*Detection limit--0.05 μg/l
†Internal standard
‡Instrument source
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 8. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT N (μg/l)*

U		Dy		Rh		Cr	680
Th		Tb		Ru		V	0.4
Bi	0.04	Gd	0.08	Mo	0.2	Ti	5.6
Pb	4.0	Eu	<0.04	Nb		Sc	<0.2
Tl		Sm	0.1	Zr	0.6	Ca	280
Hg	NR	Nd	0.1	Y	0.4	K	44
Au		Pr	0.1	Sr	0.8	Cl	3.8
Pt		Ce	0.5	Rb	0.08	S	1,240
Ir		La	0.1	Br	0.1	P	2,000
Os		Ba	14	Se	0.04	Si	2,000
Re	†	Cs		As	0.8	Al	200
W	0.04	I	0.4	Ge		Mg	110
Ta	0.003‡	Te		Ga	0.04	Na	520
Hf	<0.08	Sb	0.2	Zn	150	F	76
Lu		Sn	0.3	Cu	12	B	NR
Yb	0.08	In	†	Ni	2.9	Be	
Tm		Cd	0.08	Co	1.7	Li	0.2
Er		Ag		Ie	600		
Ho		Pd		Mn	12		

*Detection limit--0.04 μg/l
†Internal standard
‡Instrument source
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 9. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT S (μg/l)*

U	0.08	Dy	0.01	Rh		Cr	26
Th	0.21	Tb		Ru		V	6.1
Bi	2.4	Gd	0.04	Mo	6.5	Ti	40
Pb	7.1	Eu	0.05	Nb	0.1	Sc	0.14
Tl	<0.02	Sm	0.09	Zr	1.6	Ca	4,353
Hg	NR	Nd	1.0	Y	0.33	K	859
Au		Pr	0.36	Sr	6.8	Cl	129
Pt		Ce	3.4	Rb	11	S	1,882
Ir		La	4.0	Br	14	P	4,824
Os		Ba	49	Se	<0.22	Si	1,106
Re	†	Cs	0.99	As	7.0	Al	2,000
W	<0.11	I	0.38	Ge	0.16	Mg	1,035
Ta	0.55‡	Te		Ga	.56	Na	694
Hf	<0.13	Sb	188	Zn	13	F	56
Lu	<0.02	Sn	31	Cu	306	B	NR
Yb	<0.07	In	†	Ni	16	Be	<0.05
Tm	<0.02	Cd	1.1	Co	0.44	Li	0.58
Er	<0.04	Ag	0.25	Ie	2,588		
Ho	0.01	Pd		Mn	56		

*Detection limit--0.01 μg/l

†Internal standard

‡Instrument source

NR = Not reported

No data indicates "not detected above detection limit."

TABLE 10. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT T (μg/l)*

U		Dy		Rh		Cr	5.1
Th		Tb		Ru		V	0.4
Bi		Gd		Mo	0.2	Ti	7.3
Pb	53	Eu		Nb		Sc	
Tl		Sm		Zr	0.9	Ca	1,500
Hg	NR	Nd		Y	0.2	K	330
Au		Pr		Sr	1.6	Cl	11
Pt		Ce	0.4	Rb	1.1	S	140
Ir		La		Br	1.8	P	1,300
Os		Ba	150	Se		Si	1,040
Re	†	Cs		As		Al	550
W		I		Ge		Mg	530
Ta		Te		Ga		Na	2,700
Hf		Sb	0.7	Zn	98	F	890
Lu		Sn	1.1	Cu	29	B	NR
Yb		In	†	Ni	1.6	Be	
Tm		Cd		Co	0.2	Li	0.7
Er		Ag		Ie	320		
Ho		Pd		Mn	4.9		

*Detection limit--0.01 μg/l

†Internal standard

NR = Not reported

No data indicates "not detected above detection limit."

TABLE 11. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT U (μg/1)*

U	<0.02	Dy		Rh		Cr	23
Th	<0.04	Tb		Ru		V	0.49
Bi	<0.05	Gd		Mo	5.2	Ti	18
Pb	7.4	Eu	0.02	Nb		Sc	0.1
Tl	<0.02	Sm	0.02	Zr	2.2	Ca	756
Hg	NR	Nd	0.02	Y	0.16	K	402
Au		Pr	0.01	Sr	6.4	Cl	12
Pt		Ce	0.62	Rb	3.3	S	2,680
Ir		La	0.38	Br	5.5	P	597
Os		Ba	12	Se	0.09	Si	378
Re	†	Cs	0.01	As	<0.13	Al	1,220
W	<0.07	I	0.28	Ge		Mg	116
Ta	<0.23‡	Te		Ga	0.18	Na	1,950
Hf	<0.09	Sb	1.7	Zn	60	F	96
Lu		Sn	4.3	Cu	29	B	NR
Yb	<0.06	In	†	Ni	11	Be	
Tm		Cd	0.44	Co	0.20	Li	0.40
Er	<0.04	Ag	0.23	Ie	560		
Ho		Pd		Mn	12		

*Detection limit--<0.01 μg/1
†Internal standard
‡Instrument source
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 12. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT W (μg/1)*

U		Dy		Rh		Cr	10
Th	0.5	Tb	0.1	Ru		V	15
Bi	<0.2	Gd	0.5	Mo	0.5	Ti	160
Pb	14	Eu	0.2	Nb	0.2	Sc	0.4
Tl	<0.1	Sm	<0.9	Zr	1.9	Ca	15,000
Hg	NR	Nd	1.5	Y	1.1	K	1,500
Au		Pr	1.3	Sr	19	Cl	290
Pt		Ce	8.0	Rb	3.1	S	330
Ir		La	2.3	Br	2.5	P	1,700
Os		Ba	230	Se		Si	2,600
Re	†	Cs	0.1	As	13	Al	6,700
W	<0.2	I	0.07	Ge		Mg	3,700
Ta	0.006‡	Te		Ga	2.3	Na	3,300
Hf	<0.3	Sb	0.4	Zn	66	F	390
Lu		Sn	1.3	Cu	23	B	NR
Yb	<0.2	In	†	Ni	17	Be	0.2
Tm		Cd	0.3	Co	8.0	Li	13
Er	<0.1	Ag	0.07	Ie	4,500		
Ho		Pd		Mn	370		

*Detection limit--0.05 μg/1
†Internal standard
‡Instrument source
NR = Not reported
No data indicates "not detected above detection limit."

TABLE 13. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER SUSPENDED SOLIDS, PLANT X (μg/1)*

U		Dy		Rh		Cr	12
Th		Tb		Ru		V	0.4
Bi	<0.09	Gd	<0.06	Mo	0.1	Ti	4.7
Pb	5.0	Eu		Nb	0.06	Sc	0.06
Tl	<0.06	Sm	<0.06	Zr	0.4	Ca	1,200
Hg	NR	Nd	0.06	Y	0.08	K	53
Au		Pr	0.06	Sr	1.3	Cl	17
Pt		Ce	0.2	Rb	0.4	S	110
Ir		La	0.09	Br	17	P	2,400
Os		Ba	22	Se		Si	880
Re	†	Cs		As	<0.06	Al	640
W	0.06	I	0.09	Ge		Mg	
Ta	0.08‡	Te		Ga	0.06	Na	2,200
Hf	<0.1	Sb	2.4	Zn	410	F	38
Lu		Sn	0.44	Cu	27	B	NR
Yb	<0.09	In	†	Ni	7	Be	
Tm		Cd	0.06	Co	2.7	Li	0.15
Er	<0.06	Ag		Ie	1,300		
Ho		Pd		Mn	2.5		

*Detection limit--0.03 μg/1

†Internal standard

‡Instrument source

NR = Not reported

No data indicates "not detected above detection limit."

TABLE 14. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT A (mg/1)*

U	<0.003	Dy		Rh		Cr	1.4
Th	<0.003	Tb		Ru		V	2.5
Bi	<0.006	Gd	<0.003	Mo	0.043	Ti	0.087
Pb	0.38	Eu	<0.002	Nb		Sc	<0.006
Tl	<0.004	Sm	<0.002	Zr	0.031	Ca	170
Hg	NR	Nd	<0.004	Y	0.003	K	8.8
Au		Pr	0.009	Sr	0.70	Cl	11
Pt		Ce	0.002	Rb	0.19	S	130
Ir		La	0.004	Br	0.47	P	6
Os		Ba	0.51	Se	0.003	Si	18
Re	†	Cs	0.003	As	<0.015	Al	6.4
W	<0.008	I	0.011	Ge		Mg	10
Ta	0.008‡	Te		Ga		Na	180
Hf	<0.010	Sb	0.095	Zn	13	F	1.1
Lu	<0.002	Sn	0.10	Cu	0.14	B	NR
Yb	<0.007	In	†	Ni	1.0	Be	<0.003
Tm	<0.002	Cd	0.005	Co	0.021	Li	0.12
Er	<0.007	Ag	0.001	Ie	6.1		
Ho		Pd		Mn	0.48		

*Detection limit--0.001 mg/1

†Internal standard

‡Instrument source

NR = Not reported

No data indicates "not detected above detection limit."

TABLE 15. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT B (mg/1)

U	<0.003	Dy		Rh		Cr	0.24
Th	<0.003	Tb		Ru		V	0.005
Bi	<0.005	Gd	<0.003	Mo	<0.007	Ti	0.079
Pb	0.17	Eu	<0.002	Nb		Sc	<0.003
Tl	<0.002	Sm	<0.002	Zr	0.004	Ca	21
Hg	NR	Nd	<0.003	Y	0.002	K	79
Au		Pr	<0.002	Sr	0.22	Cl	2.1
Pt		Ce	≤0.003	Rb	0.25	S	300
Ir		La	≤0.002	Br	0.26	P	45
Os		Ba	0.20	Se	0.006	Si	8.0
Re	*	Cs	0.001	As	0.28	Al	0.96
W	<0.007	I	0.004	Ge		Mg	14
Ta	0.10†	Te		Ga		Na	190
Hf	<0.009	Sb	0.019	Zn	1.2	F	3.7
Lu		Sn	0.017	Cu	0.16	B	NR
Yb	<0.006	In	*	Ni	0.017	Be	<0.003
Tm	<0.002	Cd	0.006	Co	0.008	Li	0.032
Er	<0.003	Ag	0.001	Ie	2.4		
Ho		Pd		Mn	0.41		

*Internal standard
†Instrument source
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 16. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT C (mg/1)

U	<0.005	Dy	<0.003	Rh		Cr	0.053
Th	<0.005	Tb	<0.002	Ru		V	4.4
Bi	<0.010	Gd	<0.004	Mo	0.011	Ti	0.71
Pb	<0.25	Eu	<0.003	Nb	0.004	Sc	<0.017
Tl	<0.007	Sm	<0.006	Zr	0.009	Ca	41
Hg	NR	Nd	<0.005	Y	0.001	K	7.7
Au		Pr	0.004	Sr	1.1	Cl	2.3
Pt		Ce	<0.003	Rb	0.38	S	340
Ir		La	<0.004	Br	13	P	20
Os		Ba	1.1	Se	0.27	Si	27
Re	*	Cs	0.001	As	<0.025	Al	0.99
W	<0.013	I	0.024	Ge	0.004	Mg	1.8
Ta	<0.045†	Te	<0.002	Ga		Na	370
Hf	<0.017	Sb	0.62	Zn	1.2	F	12
Lu	<0.003	Sn	0.082	Cu	0.63	B	NR
Yb	<0.011	In	*	Ni	0.45	Be	<0.005
Tm	<0.003	Cd	0.030	Co	0.018	Li	0.044
Er	<0.007	Ag	10	Ie	2.1		
Ho	<0.002	Pd		Mn	0.18		

*Internal standard
†Instrument Source
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 17. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT E
(mg/1)

U		Dy		Rh		Cr	0.043
Th		Tb		Ru		V	0.018
Bi	<0.002	Gd		Mo	0.004	Ti	0.13
Pb	0.063	Eu		Nb		Sc	
Tl	<0.002	Sm	<0.002	Zr	0.009	Ca	36
Hg	NR	Nd	0.007	Y		K	26
Au		Pr	0.004	Sr	0.094	Cl	1.3
Pt		Ce	0.022	Rb	0.071	S	290
Ir		La	0.019	Br	0.92	P	9.2
Os		Ba	0.29	Se	0.002	Si	2.9
Re	*	Cs		As	0.035	Al	0.39
W	<0.003	I	0.011	Ge		Mg	2.1
Ta	0.001†	Te		Ga		Na	70
Hf	<0.003	Sb	0.13	Zn	0.76	F	13
Lu		Sn	0.006	Cu	0.10	B	NR
Yb	<0.002	In	*	Ni	0.038	Be	
Tm		Cd	0.004	Co	0.001	Li	0.014
Er	<0.002	Ag	0.001	Ie	0.86		
Ho		Pd		Mn	0.035		

*Internal standard
†Instrument source
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 18. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT F
(mg/1)

U	<0.003	Dy	<0.002	Rh		Cr	0.015
Th	<0.004	Tb		Ru		V	0.022
Bi	<0.007	Gd	<0.003	Mo	0.005	Ti	0.10
Pb	0.033	Eu	<0.002	Nb		Sc	<0.011
Tl	<0.005	Sm	<0.004	Zr	0.011	Ca	27
Hg	NR	Nd	<0.004	Y		K	10
Au		Pr	0.002	Sr	0.28	Cl	0.83
Pt		Ce	0.003	Rb	0.054	S	56
Ir		La	0.002	Br	0.16	P	38
Os		Ba	0.16	Se	0.006	Si	48
Re	*	Cs		As	<0.017	Al	4.6
W	<0.009	I	0.002	Ge		Mg	15
Ta	<0.003†	Te	<0.002	Ga	0.001	Na	490
Hf	<0.011	Sb	0.19	Zn	1.5	F	2.9
Lu	<0.002	Sn	0.004	Cu	0.49	B	NR
Yb	<0.007	In	*	Ni	0.024	Be	<0.003
Tm	<0.002	Cd	0.004	Co	0.021	Li	0.004
Er	<0.005	Ag		Ie	2.7		
Ho		Pd		Mn	0.13		

*Internal standard
†Instrument source
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 19. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT G (mg/l)

U		Dy		Rh		Cr	0.018
Th		Tb		Ru		V	0.12
Bi		Gd		Mo	0.006	Ti	0.11
Pb	0.10	Eu		Nb		Sc	
Tl		Sm		Zr	0.011	Ca	16
Hg	NR	Nd		Y		K	20
Au		Pr		Sr	0.089	Cl	1.1
Pt		Ce	0.004	Rb	0.017	S	43
Ir		La	0.004	Br	0.036	P	16
Os		Ba	0.23	Se	0.001	Si	8.7
Re	*	Cs	0.001	As	0.014	Al	2.2
W		I	0.007	Ge		Mg	1.4
Ta		Te		Ga		Na	48
Hf		Sb	1.2	Zn	0.84	F	3.2
Lu		Sn	0.032	Cu	0.11	B	NR
Yb		In	*	Ni	0.038	Be	
Tm		Cd	0.002	Co	0.13	Li	0.32
Er		Ag	0.003	Ie	1.6		
Ho		Pd		Mn	0.17		

*Internal standard
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 20. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT K (mg/l)

U	<0.002	Dy		Rh		Cr	0.090
Th	<0.002	Tb		Ru		V	0.015
Bi	<0.004	Gd	<0.002	Mo	0.006	Ti	0.018
Pb	0.017	Eu	≤0.002	Nb		Sc	<0.002
Tl		Sm	≤0.002	Zr	0.005	Ca	24
Hg	NR	Nd	<0.002	Y		K	26
Au		Pr		Sr	0.19	Cl	650
Pt		Ce		Rb	0.027	S	105
Ir		La	<0.002	Br	2.9	P	6.8
Os		Ba	0.19	Se		Si	120
Re	*	Cs		As	0.32	Al	0.71
W	<0.005	I	23	Ge	0.010	Mg	13
Ta	<0.015†	Te		Ga	0.001	Na	120
Hf	<0.006	Sb	0.048	Zn	0.77	F	87
Lu		Sn	0.011	Cu	0.11	B	NR
Yb	<0.004	In	*	Ni	0.014	Be	<0.002
Tm		Cd	0.003	Co	0.012	Li	0.005
Er	<0.003	Ag		Ie	0.72		
Ho		Pd		Mn	0.008		

*Internal standard
†Instrument source
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 21. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT L (mg/1)

U		Dy		Rh		Cr	0.26
Th		Tb		Ru		V	0.23
Bi	<0.002	Gd		Mo	0.015	Ti	0.24
Pb	0.14	Eu		Nb	0.005	Sc	
Tl		Sm	≤0.007	Zr	0.026	Ca	110
Hg	NR	Nd		Y	0.001	K	5.3
Au		Pr		Sr	0.99	Cl	2.1
Pt		Ce	0.011	Rb	0.20	S	330
Ir		La	0.005	Br	0.51	P	10
Os		Ba	0.37	Se	0.002	Si	15
Re	*	Cs	0.007	As	0.031	Al	0.44
W	<0.003	I	0.005	Ge		Mg	4.6
Ta	0.003†	Te		Ga		Na	78
Hf	<0.004	Sb	0.30	Zn	2.4	F	2.5
Lu		Sn	0.046	Cu	0.54	B	NR
Yb	<0.003	In	*	Ni	0.25	Be	
Tm		Cd	0.003	Co	0.033	Li	2.1
Er	0.001	Ag	0.001	Ie	4.5		
Ho		Pd		Mn	0.27		

*Internal standard
†Instrument source
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 22. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT N (mg/l)

U	<0.004	Dy	<0.002	Rh		Cr	44
Th	<0.004	Tb		Ru		V	0.033
Bi	<0.008	Gd	<0.003	Mo	0.030	Ti	0.089
Pb	0.95	Eu	<0.003	Nb	0.001	Sc	<0.004
Tl	<0.002	Sm	<0.005	Zr	0.054	Ca	570
Hg	NR	Nd	<0.004	Y	0.017	K	58
Au		Pr	0.002	Sr	2.1	Cl	1.1
Pt		Ce	0.005	Rb	0.51	S	1,400
Ir		La	0.008	Br	0.19	P	110
Os		Ba	1.3	Se	0.063	Si	54
Re	*	Cs	0.001	As	0.40	Al	110
W	<0.010	I	0.12	Ge	0.005	Mg	42
Ta	<0.011	Te		Ga	0.002	Na	150
Hf	<0.012	Sb	0.12	Zn	580	F	41
Lu	<0.002	Sn	0.008	Cu	0.11	B	NR
Yb	<0.008	In	*	Ni	0.39	Be	<0.004
Tm	<0.003	Cd	0.004	Co	0.46	Li	0.033
Er	<0.004	Ag	0.002	Ie	80		
Ho		Pd		Mn	27		

*Internal standard
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 23. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT S
(mg/l)

U	<0.002	Dy		Rh		Cr	0.016
Th	<0.002	Tb	≤0.002	Ru		V	0.006
Bi	0.012	Gd		Mo	0.021	Ti	0.010
Pb	0.085	Eu		Nb	0.002	Sc	<0.002
Tl		Sm		Zr	0.016	Ca	11
Hg	NR	Nd	≤0.004	Y		K	73
Au		Pr	≤0.005	Sr	0.027	Cl	2.5
Pt		Ce	≤0.005	Rb	0.81	S	24
Ir		La	≤0.007	Br	13	P	15
Os		Ba	0.097	Se	0.001	Si	18
Re	*	Cs	0.002	As	0.064	Al	18
W	<0.005	I	0.017	Ge		Mg	2.8
Ta	<0.012	Te		Ga		Na	95
Hf	<0.004	Sb	0.84	Zn	0.29	F	1.1
Lu		Sn	0.024	Cu	0.28	B	NR
Yb	<0.003	In	*	Ni	0.005	Be	<0.002
Tm	<0.002	Cd	0.008	Co		Li	0.011
Er	≤0.003	Ag	0.001	Ie	1.0		
Ho		Pd		Mn	0.10		

*Internal standard
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 24. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT T
(mg/l)

U	0.002	Dy		Rh		Cr	0.058
Th		Tb		Ru		V	0.014
Bi		Gd		Mo	0.005	Ti	0.019
Pb	0.042	Eu		Nb		Sc	
Tl		Sm		Zr	0.006	Ca	5.2
Hg	NR	Nd		Y	0.002	K	36
Au		Pr	0.002	Sr	0.030	Cl	0.51
Pt		Ce	0.002	Rb	0.13	S	0.70
Ir		La	0.006	Br	0.13	P	0.79
Os		Ba	0.022	Se	0.005	Si	1.9
Re	*	Cs	0.001	As	<0.003	Al	3.3
W	<0.002	I	0.002	Ge		Mg	1.4
Ta		Te		Ga		Na	40
Hf	<0.002	Sb	0.009	Zn	0.29	F	0.95
Lu		Sn	0.005	Cu	0.040	B	NR
Yb	<0.002	In	*	Ni	0.045	Be	
Tm		Cd	0.001	Co	0.002	Li	0.008
Er	<0.002	Ag	0.002	Ie	0.57		
Ho	≤0.002	Pd		Mn	0.059		

*Internal standard
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 25. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT U
(mg/1)

U		Dy		Rh		Cr	0.005
Th		Tb		Ru		V	0.002
Bi	<0.002	Gd		Mo	0.007	Ti	0.024
Pb	0.006	Eu		Nb		Sc	0.003
Tl		Sm		Zr	0.002	Ca	180
Hg	NR	Nd		Y	0.001	K	37
Au		Pr	≤0.026	Sr	0.32	Cl	170
Pt		Ce		Rb	0.043	S	16
Ir		La	≤0.081	Br	0.55	P	6.4
Os		Ba	0.16	Se	0.018	Si	14
Re	*	Cs	0.002	As	0.14	Al	0.24
W	<0.002	I	0.076	Ge		Mg	11
Ta		Te		Ga		Na	83
Hf	<0.002	Sb	0.19	Zn	16	F	2.4
Lu		Sn	0.003	Cu	0.099	B	NR
Yb		In	*	Ni	0.058	Be	
Tm		Cd	0.003	Co	0.10	Li	0.023
Er		Ag		Ie	0.12		
Ho		Pd		Mn	0.53		

*Internal standard
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 26. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT V
(mg/1)

U		Dy		Rh		Cr	0.066
Th		Tb		Ru		V	0.012
Bi	0.003	Gd		Mo	0.005	Ti	0.12
Pb	0.089	Eu		Nb		Sc	
Tl		Sm		Zr	0.002	Ca	43
Hg	NR	Nd	≤0.002	Y	0.001	K	2.0
Au		Pr	≤0.002	Sr	0.19	Cl	1.0
Pt		Ce	0.005	Rb	0.022	S	45
Ir		La	0.003	Br	1.1	P	4.2
Os		Ba	0.36	Se		Si	9.5
Re	*	Cs		As	0.012	Al	4.0
W	<0.002	I	≤0.003	Ge		Mg	7.4
Ta		Te		Ga		Na	42
Hf	<0.002	Sb	0.010	Zn	3.1	F	1.4
Lu		Sn	0.009	Cu	2.0	B	NR
Yb		In	*	Ni	0.010	Be	
Tm	≤0.001	Cd	0.011	Co	0.073	Li	0.041
Er	≤0.002	Ag		Ie	4.7		
Ho		Pd		Mn	0.31		

*Internal standard
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 27. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT W (mg/l)

U	<0.003	Dy	≤0.001	Rh		Cr	0.028
Th	≤0.003	Tb		Ru		V	0.011
Bi	≤0.005	Gd	<0.002	Mo	<0.007	Ti	0.079
Pb	0.17	Eu	≤0.004	Nb		Sc	<0.002
Tl	<0.002	Sm	≤0.003	Zr	0.023	Ca	94
Hg	NR	Nd	≤0.004	Y	0.006	K	660
Au		Pr	≤0.004	Sr	0.31	Cl	1.2
Pt		Ce	≤0.010	Rb	2.0	S	16
Ir		La	≤0.006	Br	0.55	P	0.12
Os		Ba		Se	0.018	Si	30
Re	*	Cs	0.003	As	0.033	Al	7.3
W	<0.007	I	≤0.013	Ge	<0.004	Mg	12
Ta	<0.003†	Te		Ga	0.004	Na	8.3
Hf	≤0.009	Sb	≤0.013	Zn	0.060	F	11
Lu	≤0.001	Sn	0.011	Cu	0.072	B	NR
Yb	<0.006	In	*	Ni	0.024	Be	<0.003
Tm	<0.002	Cd	0.006	Co	0.048	Li	0.054
Er	<0.003	Ag	0.003	Fe	0.67		
Ho		Pd		Mn	0.22		

*Internal standard
†Instrument source
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 28. SPARK SOURCE MASS SPECTROSCOPY--WASTEWATER FILTRATE, PLANT X (mg/l)

U		Dy		Rh		Cr	0.027
Th		Tb		Ru		V	0.026
Bi		Gd		Mo	0.005	Ti	0.018
Pb	0.030	Eu		Nb		Sc	<0.002
Tl		Sm		Zr		Ca	23
Hg	NR	Nd		Y	0.007	K	9.0
Au		Pr		Sr	0.14	Cl	130
Pt		Ce		Rb	0.023	S	2.9
Ir		La	0.001	Br	2.5	P	29
Os		Ba	0.13	Se		Si	18
Re	*	Cs		As	0.022	Al	8.2
W	<0.002	I	0.037	Ge	0.005	Mg	4.6
Ta		Te		Ga	0.001	Na	33
Hf	<0.002	Sb	1.9	Zn	0.14	F	11
Lu		Sn	0.004	Cu	0.87	B	NR
Yb	<0.002	In	*	Ni	0.006	Be	-
Tm		Cd		Co	0.12	Li	0.052
Er		Ag		Fe	2.5		
Ho		Pd		Mn	0.028		

*Internal standard
NR = Not reported
No data indicates "not detected above detection limit of 0.001 mg/l."

TABLE 29. ATOMIC ABSORPTION (AA)--WET CHEMICAL METHODS RAW WASTE AND SECONDARY EFFLUENTS (mg/1)

(Secondary Effluent Data in Parentheses)

Plant	Hg		Sb		As	
A	0.004	(†)	†	(0.03)	†	(†)
B	0.0009	(0.0006)	†	(†)	†	(†)
C	†	(0.0007)	0.007	(0.004)	†	(†)
D	†	(†)	0.003	(0.002)	0.017	(0.006)
E	†	(†)	0.008	(0.0008)	†	(†)
F	†	(0.0009)	0.001	(0.0003)	†	(†)
G	†	(†)	0.052	(0.011)	†	(†)
H	†	(†)	0.004	(0.006)	†	(†)
J	†	(†)	0.0007	(†)	†	(†)
K	†	(†)	0.003	(0.0008)	0.006	(†)
L	†	(†)	0.005	(0.003)	†	(†)
M	†	(†)	0.0008	(0.004)	†	(†)
N	0.0004	(†)	0.0002	(0.002)	†	(†)
P	†	(†)	†	(†)	†	(†)
R‡	†	(†)	†	(†)	†	(†)
S	†	(†)	0.057	(0.074)	0.005	(†)
T	0.0007	(†)	-		†	(†)
U	0.0004	(†)	0.007	(0.001)	†	(†)
V	†	(†)	†	(0.004)	†	(†)
W	†	(0.0005)	†	(†)	†	(0.004)
X	†	(0.0009)	0.0003	(0.0009)	†	(†)
Y	†	(†)	0.016	(0.003)	†	(†)
Z	†	(†)	0.011	(0.012)	†	(†)
Y-001	§	(§)	†	(†)	†	(†)
C-001	§	-	§	-	†	-
JJ	§	(§)	§	(§)	0.20	(0.16)
KK	§	(§)	§	(§)	0.12	(†)
LL	§	(§)	§	(§)	0.10	(0.07)
MM-1	§	-	§	-	0.055	
MM-2	§	-	§	-	0.003	
MM-3	§	-	§	-	0.007	
MM-4	§	-	§	-	0.006	
NN	§	(§)	§	(§)	†	(†)
OO	§	(§)	§	(§)	†	(†)
PP	§	-	§	-	†	-

* Sb by ICAP.
† Concentration below detection limit of 0.0005 for Hg and Sb and 0.005 for As.
‡ Secondary effluent inadvertently collected prior to settling pond.
§ Analysis not performed.
- No data reported.

TABLE 30. AQUEOUS ANALYSES--RAW WASTE

Sample description		pH	Acidity	Alkalinity	BOD mg/1	COD mg/1	D.O.	Conductivity	Dissolved solids	Suspended solids mg/1	Anions mg/1 CN
Plant	A	10.7			459	1,735				165	<.004
	B	10.5			1,050	1,264				32	.017
	C	11.2			445	802				49	.007
	D	10			71	224				16	.21
	E	10			18	2,660				52	<.004
	F	9.2			194	583				23	<.004
	G	11			203	1,340				37	<.004
	H	10			288	320				39	<.004
	J	11			210	810				0.01	<.004
	K	10			564	1,725				69	<.004
	L	7.4			379	1,117				19	<.004
	M	11			830	2,265				210	<.004
	N	9.2			334	1,140				68	<.004
	P	10			680	172				6	.19
	R	10			450	1,692				87	<.004
	S	10			219	559				25	.007
	T	9.5			501	500				28	<.004
	U	10			400	1,464				111	<.004
	V	9.0			53	*				54	.006
	W	10.4			1,920	6,124				2,300	.015
	X	10.2			237	786				24	<.004
	Y	10.5			122	457				33	<.004
	Z	10			351	812				20	<.004
	JJ	*			*	1,545				*	.005

TABLE 30 (con.)

Sample description	pH	Acidity	Alkalinity	BOD mg/1	COD mg/1	D.O.	Conductivity	Dissolved solids	Suspended solids mg/1	Anions mg/1 CN
Plant KK	*			*	1,955				*	<.004
LL	*			*	727				*	.008
MM-1	*			*	*				*	<.004
MM-2	*			*	*				*	<.004
MM-3	*			*	*				*	<.004
MM-4	*			*	*				*	<.004
NN	*			938	*				*	.04
OO	*			1,889	*				*	<.004
Y-001	*			*	*				*	<.004

* Analysis not performed on sample.

TABLE 31. AQUEOUS ANALYSES--SECONDARY EFFLUENT

Sample description	pH	Acidity	Alkalinity	BOD mg/1	COD mg/1	D.O.	Conductivity	Dissolved solids	Suspended solids mg/1	Anions mg/1 CN
Plant A	7.3			168	1,652				228	.015
B	7.5			< 5	99				8	<.004
C	10			25	396				300	.013
D	7.2			6.6	64				154	.21
E	7.2			< 5	78				19	<.004
F	7.4			69	276				44	<.004
G	7.5			42	502				6	.006
H	7.6			14	300				43	<.004
J	7.8			25	376				0.023	<.004
K	7.2			< 5	131				21	<.004
L	5.8			13	234				78	.172
M	7.5			< 5	255				21	<.004
N	7.0			36	286				77	<.004
P	7.1			28	45				45	.14
R	8.1			70	830				225	<.004
S	7.8			59	1,035				581	<.004
T	7.4			32	414				35	<.004
U	7.3			24	748				92	.212
V	7.1			< 5	128				26	.018

TABLE 31 (con.)

Sample description	pH	Acidity	Alkalinity	BOD mg/l	COD mg/l	D.O.	Conductivity	Dissolved solids	Suspended solids mg/l	Anions mg/l CN
Plant W	8.1			84	837				300	.020
X	7.2			15	258				18	.101
Y	8.0			< 5	115				17	<.004
Z	8.0			< 5	105				13	<.004
JJ	*			*	510				*	<.028
KK	*			*	447				*	<.004
LL	*			*	155				*	.006
MM-1	*			*	*				*	<.004
MM-2	*			*	*				*	<.004
MM-3	*			*	*				*	<.004
MM-4	*			*	*				*	<.004
NN	*			*	236				*	<.004
OO	*			*	635				*	<.004
PP	*			*	339				*	<.004
Y-001	*			*	*				*	.029
C-001	*			*	*				*	<.004

* Analysis not performed on sample.

TABLE 32. AQUEOUS ANALYSES--SECONDARY EFFLUENT FOR LEVEL I ANALYSIS

Sample description	pH	Acidity mg/l (Methyl Orange)	Alkalinity mg/l	BOD mg/l	COD mg/l	D.O. mg/l	Conductivity µmhos @ 25° C	Dissolved solids mg/l	Suspended solids mg/l	Anions mg/l				
										Cr	SO_3	NO_2	NO_3	H_2S
PLANT A	7.3	0	100	168	1,652	5.5	1,500	1,725	234	0.18	8.5	0.06	1.9	4
B	7.5	0	5.5	NA	99	7	1,200	1,681	7	0.004	368	<0.005	0.002	0.20
C	10	0	8.3	25	396	6	2,400	2,924	24	0.031	40	4.64	23.3	5
E	7.5	0	35	< 5	78	8.5	310	13,120	10	0.004	12	0.016	79.2	0.1
F	7.4	0	2.4	69	276	5	1,900	2,006	10	0.004	10	0.043	0	0.1
G	7.5	0	30	42	502	8	155	276	5	0.003	56	0.076	1.32	<2
K	7.2	0	710	< 5	131	5	875	1,256	14	0.004	>1	0.056	4.4	2
L	5.8	0	30	13	234	4	555	725	42	0.03	460	0.864	13.5	3
M	7.0	20	0	36	286	9	990	1,352	13	1.8	640	0.003	5.5	0.1
S	7.8	0	130	59	1,035	7	640	692	349	0	150	0.033	4.4	0.3
T	7.4	0	300	32	414	8	460	660	44	0	100	0.04	0.8	6
U	7.3	0	120	24	748	9	770	1,331	111	0.014	0	<0.005	0.8	3.5
V	7.1	0	0.4	< 5	128	9	360	NA	NA	0.003	57	0.264	0.88	0.5
W	8.1	0	950	84	837	5	1,250	1,648	217	0.003	0	0.145	12.3	0.1
X	7.2	0	140	15	258	7.2	285	437	1.3	0.039	1	0.44	0.033	0.01

NA - Not analyzed.
Samples for D.O., pH, color and anions were not extracted prior to analysis.

TABLE 33. LC FRACTIONATION*--EXTRACTS OF SECONDARY EFFLUENTS

	Plant A				Plant B			
Fraction	TCO	Grav mg/l	Total	Total	TCO	Grav mg/l	Total	Total
LC 1		<0.01				<0.01		
LC 2		1.61				1.03		
LC 3		0.53				<0.01		
LC 4		<0.01				0.38		
LC 5		<0.01				<0.01		
LC 6		1.02				1.39		
LC 7		<0.01				<0.01		
LC 8†		2.10				1.50		

	Plant F				Plant G			
Fraction	TCO	Grav mg/l	Total	Total	TCO	Grav mg/l	Total	Total
LC 1		0.94				3.18		
LC 2		0.03				0.72		
LC 3		0.19				0.23		
LC 4		0.33				0.09		
LC 5		0.14				0.03		
LC 6		1.86				2.90		
LC 7		5.17				1.39		
LC 8†		4.27				1.55		

See footnotes at end of table.

TABLE 33 (con.)

	Plant L				Plant N			
Fraction	TCO	Grav mg/l	Total	Total	TCO	Grav mg/l	Total	Total
LC 1		1.27				4.45		
LC 2		0.22				0.10		
LC 3		0.86				0.39		
LC 4		0.86				0.21		
LC 5		0.97				0.10		
LC 6		5.51				2.05		
LC 7		11.7				0.89		
LC 8†		1.81				1.66		

	Plant S				Plant T			
Fraction	TCO	Grav mg/l	Total	Total	TCO	Grav mg/l	Total	Total
LC 1		1.23				1.85		
LC 2		0.47				0.26		
LC 3		1.97				0.67		
LC 4		1.11				1.29		
LC 5		0.66				0.69		
LC 6		2.68				1.74		
LC 7		0.65				2.92		
LC 8†		1.36				1.71		

See footnotes at end of table.

TABLE 33 (con.)

Fraction	Plant U				Plant V			
	TCO	Grav mg/l	Total	Total	TCO	Grav mg/l	Total	Total
LC 1		19.7				1.53		
LC 2		2.7				0.49		
LC 3		3.67				0.38		
LC 4		1.47				0.55		
LC 5		1.77				1.18		
LC 6		7.3				0.39		
LC 7		3.83				2.26		
LC 8†		7.93				1.98		

Fraction	Plant W			
	TCO	Grav mg/l	Total	Total
LC 1		2.09		
LC 2		0.34		
LC 3		1.32		
LC 4		0.85		
LC 5		0.57		
LC 6		0.42		
LC 7		1.41		
LC 8†		1.17		

*TCO was not determined

†Weights for fraction 8 contain silica contaminant; blank value not subtracted from total.

TABLE 34. IR REPORT
SAMPLE: ORGANIC EXTRACTS BEFORE LC

Plant	
A	Bonded OH, aliphatic CH, C=O ester and acid, ketone or aldehyde, conjugated C=C, possible aromatic C=C, ether groups, $(CH_2)n$, where $n \geq 4$.
B	Bonded OH, aliphatic CH, ether $(CH_2)_4$, C=0, or C=C?
E	Aliphatic CH, diffuse C=0 region, carboxylate ion, ether group complex spectrum.
F	Bonded OH, aliphatic CH, diffuse C=0, and C=C regions, Si-0 possible, trace CH_2Cl_2.
G	Aromatic and aliphatic CH, residual CH_2Cl_2 in spectrum.
K	Bonded OH, aliphatic CH, diffuse C=0 region $Si(CH_3)$ group? Diffuse spectrum.
L	Bonded OH, aliphatic CH, C=0, aromatic or conjugated C=C, ether group. Complex spectrum C≡N, SiO_2 -?
N	Bonded OH, aliphatic CH, diffuse C=0, C=C region, silicone $(CH_2)_4$ ether?
S	Bonded OH, aliphatic CH, ester C=0, $(CH_2)n$ groups, broad diffuse spectrum, ether groups possible.
T	Bonded OH, aliphatic CH, C=0, C=C, ether or SiO groups, $(CH_2)n$ groups. Very diffuse spectrum.
U	Trace bonded OH, aliphatic CH, acid, ketone or aldehyde C=0, silicones, some $(CH_2)n$ groups.
V	Trace bonded OH, aliphatic CH, ester C=0, acid, aldehyde or ketone C=0, silicone adsorption, some $(CH_2)n$ groups.
W	Bonded OH, aliphatic CH, ester C=0, acid, ketone, or aldehyde C=0. $(CH_2)n$ - n where $n > 4$, diffuse spectrum 1,300 cm^{-1} to 900 cm^{-1}.
X	Bonded OH, aliphatic CH, C=0, ether or SiO, $(CH_2)n$, numerous broad diffuse bonds.

TABLE 35. IR REPORT
SAMPLE: PLANT A

LC 1	All aliphatic hydrocarbons.
LC 2	Aliphatic and aromatic hydrocarbons, aromatic C=C.
LC 3	Aliphatic C-H, ester or aldehyde C=O, conjugated C=C, various CH_2 groups, or aromatic substitution bonds.
LC 4	Similar to fraction 3.
LC 5	Similar to fraction 3.
LC 6	Bonded OH, aliphatic C-H, acid, ketone or aldehyde C=O, conjugated and aromatic C=C, possible phthalate ester, ether group or Si-O.
LC 7	Bonded OH, aliphatic CH, ester or aldehyde C-O, water in material, ether or Si-O groups.
LC 8	Bonded OH and 1,630 cm^{-1} absorption - water, aliphatic C-H, trace of C=O, SiO_2, poorly defined organic.

TABLE 36. IR REPORT
SAMPLE: PLANT B

LC 1	Aliphatic CN, trace aromatic CH, Si-CH_3 (?), methylene CH_2 groups >4.
LC 2	Very strong background adsorption--only aliphatic CH visible.
LC 3	Bonded OH, aliphatic CH, ester C=O, acid, aldehyde or ketone C=O $(CH_2)n$ or >4.
LC 4	Bonded OH, trace aromatic CH, aliphatic CH, ester C=O, acid, aldehyde or ketone C=O, various CH_2 groups.
LC 5	Similar to 4, but stronger bonded OH, less ester C=O, more acid, aldehyde or ketone C=O, various CH_2 groups, complex spectrum.
LC 6	Bonded OH, aliphatic CH, some ester C=O, acid, aldehyde or ketone C=O, ether groups, may contain glycol ether type compounds.
LC 7	Bonded OH, aliphatic CH, ester C=O, acid, aldehyde or ketone C=O, strong ether group, glycol ether type of compound.
LC 8	Strong bonded OH, weak C-H (aliphatic) trace C=O, nonconjugated C=C, SiO_2 present.

TABLE 37. IR REPORT
SAMPLE: PLANT E

LC 1	Aliphatic hydrocarbons, no indication of number of CH_2 groups.
LC 2	Poor spectrum--aliphatic CH, CO_2, and water vapor in spectrum.
LC 3	Poorly defined spectrum--aliphatic CH, numerous ill-defined bonds, no C=O or C=C.
LC 4	Aliphatic CH, ester C=O, strong background adsorption.
LC 5	Aliphatic CH, ester =O, most likely aliphatic ester, possibly acetate--may be single compound.
LC 6	Spectrum too strong--bonded OH, aliphatic CH, ester C=O, acid, ketone or aldehyde C=O, large portion of compound in No. 5 plus additional carbonyl compounds.
LC 6	(Repeat) Mixture of several compounds, ester C=O, and acid, ketone or aldehyde C=O, ether group.
LC 7	Bonded OH, aliphatic CH, acid, ketone or aldehyde C=O, spectrum too strong for good ID.
LC 7	(Repeat) Bonded OH, aliphatic CH, acid, aldehyde or ketone C=O, evidence of both acid and acid salt (carboxylate ion) $CH(CH_3)_2$ group possible, no long chain $(CH_2)_n$ groups.
LC 8	Bonded OH - evidence of water (3,350 cm^{-1} and 1,635 cm^{-1}), SiO_2 present, aliphatic CH.

TABLE 38. IR REPORT
SAMPLE: PLANT F

LC 1	Long chain aliphatic hydrocarbons, unknown 1,265 cm^{-1} bond, Si-(CH_3)- ?
LC 2	Aliphatic CH, ester C=0, conjugated C=C, trace 1,265 cm^{-1}.
LC 3	Weak bonded OH, aliphatic CH, aromatic CH-?, ester C=0, conjugated C=C, progression of CH substitution bonds, series of CH_2 bond.
LC 4	Similar to No. 3, series of ill-defined bonds below 1,400 cm^{-1}.
LC 5	Bonded OH, trace aromatic CH, aliphatic CH, ester or aldehyde C=0, conjugated C=C, series of CH_2 groups aromatic substitution bonds - ?
LC 6	Bonded OH, aliphatic CH, ester C=0, nonconjugated C=C or amides, ether or SiO groups, secondary amide possible, CH_2 groups n >4.
LC 7	Similar to No. 6.
LC 8	Strong bonded OH, aliphatic CH, weak ester or aldehyde C=0, H_2O present (1,640 cm^{-1}), SiO_2 present.

TABLE 39. IR REPORT
SAMPLE: PLANT G

LC 1	Aliphatic hydrocarbon--chain length >C_4, possible $C(CH_3)_3$ group.
LC 2	Aliphatic CH, ester C=O, phthalate bonds, various chain lengths of CH_2.
LC 3	Aliphatic CH, ester C=O, some C=C, various CH_2 groupings.
LC 4	Bonded O-H, aliphatic CH, ester C=O, some acid, aldehyde or ketone C=O, C=C, possible fatty acid group, various CH_2 groups.
LC 5	Identical to No. 4.
LC 6	Considerable bonded OH, aliphatic CH, ester C=O, conjugated C=C, Si-O or ether group.
LC 7	Similar to No. 6.
LC 8	Considerable O-H, aliphatic C-H, ester C=O, SiO or ether groups.

TABLE 40. IR REPORT
SAMPLE: PLANT K

LC 1	Appears to contain water, aliphatic C-H, $Si-CH_3$, mainly hydrocarbon compounds.
LC 2	Aliphatic hydrocarbons, silicones.
LC 3	Mainly silicone type material.
LC 4	Bonded OH, some aromatic CN, aliphatic CH, ester C=O, acid, ketone or aldehyde C=O, carboxylic ion, conjugated C=C, some silicone material, $(CH_2)n$ where $n \geq 4$.
LC 5	Strong background adsorption, aliphatic CH, ester C=O various $(CH_2)n$ groups, some silicone adsorption.
LC 6	Bonded OH, aliphatic CH, trace aromatic CH, ester C=O, aromatic C=C, silicone adsorption.
LC 7	Similar to No. 6, not as strong a spectrum.
LC 8	Strong OH adsorption, very weak C-H, trace of C=O (may be H_2O background) SiO_2 adsorption, low organic content.

TABLE 41. IR REPORT
SAMPLE: PLANT L

LC 1	Aliphatic CH, trace C=O, C=C, CH_2 group >4, possible CH $(CH_3)_2$ group--mainly aliphatic hydrocarbons.
LC 2	Aliphatic hydrocarbons--branched chain, trace of C=O, C=C no long $(CH_2)n$ groups.
LC 3	Bonded OH, aliphatic CH, ester C=O, conjugated C=C strong ether group, possible glycol ether.
LC 4	Bonded OH, aliphatic CH, ester C=O, nitrite (C≡O, group, strong ether group, various straight and branched CH_2 groups.
LC 5	Bonded OH, some aromatic CH, aliphatic CH, C≡N nitrite group, ester C=O, conjugated or aromatic C=C, ether group, very complex mixture spectrums.
LC 6	Similar to No. 5 but less C≡N, more bonded OH, ester C=O, conjugated or aromatic C=C, some aromatic C-H, aliphatic CH, ether grouping--complex spectra.
LC 7	Some bonded OH, aliphatic CH, trace C≡H, carboxylate ion, possible C-Cl group.
LC 8	Contains water and SiO_2, plus some of materials found in No. 7--Carboxylate ion.

TABLE 42. IR REPORT
SAMPLE: PLANT N

LC 1	Long chain aliphatic hydrocarbons.
LC 2	Aliphatic CH, ester C=0, possible $CH(CH_3)_2$ group, spectrum not very distinct.
LC 3	Weak bonded OH, aliphatic CH, ester C=0, conjugated C=C, $CH(CH_3)_2$ group, various CH_2 groups.
LC 4	Weak bonded OH, aliphatic CH, ester C=0, possible fatty acid groups.
LC 5	Bonded OH, aliphatic CH, medium ester C=0, nonconjugated C=C, not very distinct spectrum.
LC 6	Bonded OH, aliphatic CH, ester C=0, acid, aldehyde or ketone C=0, conjugated C=C, aliphatic ketone or ester group, possible ether group, various CH_2 chain length.
LC 7	Bonded OH, aliphatic CH, ester C=0, conjugated C=C, possible fatty acid groups, ether group.
LC 8	Strong OH, weak aliphatic CH, ketone, acid, or aldehyde C=0, strong C=C, ether group.

TABLE 43. IR REPORT
SAMPLE: PLANT S

LC 1	Aliphatic hydrocarbon, chain length < C_4, unidentified bond at 1,265 cm^{-1}.
LC 2	Aliphatic hydrocarbons, trace of ester C=0, trace of C=C, not well defined below 1,300 cm^{-1}.
LC 3	Aliphatic CN, trace of aromatic CH, very strong ester C=0 or aldehyde C=0. Ester group may be acetate; if aldehyde, long chain aldehyde.
LC 4	Strong background adsorption 4,000 cm^{-1} to 900 cm^{-1}. Aliphatic CH, ester or aldehyde C=0. Poor spectrum for interpretation.
LC 5	Nearly identical to No. 4.
LC 6	Very complex spectra. Bonded OH, aliphatic CH, ester and acid, ketone or aldehyde C=0, ether or SiO groups. Several types of CH_2 groupings.
LC 7	Weak and diffuse spectra. Aliphatic CH, ester C=0 serve C=C, ether or SiO group.
LC 8	Strong OH group, weak aliphatic CH, weak acid; aldehyde or ketone C=0, strong conjugated C=C contains some SiO_2, diffuse spectrum.

TABLE 44. IR REPORT
SAMPLE: PLANT T

LC 1	Bonded OH, aliphatic CH, ester C=0, some C=C, CH_2 groups $>C_4$.
LC 2	Bonded OH, aliphatic CH, medium ester C=0, possible fatty acid group, no definite CH_2 groupings.
LC 3	Bonded OH, aliphatic CH, strong ester C=P, nonconjugated C=C, ether or SiO groups. Various chain lengths of $(CH_2)n$.
LC 4	Bonded OH, medium aliphatic CH, medium ester C=0, nonconjugated C=C, ether or SiO group, various $(CH_2)n$ groups, possible acid salts.
LC 5	Similar to No. 3.
LC 6	Bonded OH, aliphatic CH, medium ester C=0, series of 5 unknown bonds, medium intensity 1,510 cm^{-1} to 1,610 cm^{-1}, ether or SiO group, various $(CH_2)n$ groups.
LC 7	Similar to No. 6, but weaker OH, C=CO, conjugated C=C, fatty acid groups? Ether or SiO group. $(CH_2)n$ groups.
LC 8	Very strong bonded group, medium aliphatic CH, weak ester C=0, strong nonconjugated C=C possible fatty acid group, ether or SiO group, no $CH_2 > C_4$.

TABLE 45. IR REPORT
SAMPLE: PLANT U

LC 1	Aliphatic hydrocarbon, conjugated or aromatic C=C, weak, $(CH_2)n$--no > 4.
LC 2	Aliphatic hydrocarbon, conjugated C=C (alkene?) diffuse CH_2 groups.
LC 3	Aliphatic C-H, ester C=O, conjugated or aromatic C=C, possible unsaturated ester-fumarate, maleate, etc.
LC 4	Bonded OH, trace sec N-H or oxcetone C=O, aliphatic C-H, possible trace C≡N, ester C=O, conjugated C=C, unsaturated ester group. Complex spectra below 1,500 cm^{-1}.
LC 5	Aliphatic C-H, ester C=O, diffuse spectra below 1,100 cm^{-1}.
LC 6	Complex spectra, bonded OH, NH or C=O overtone, aliphatic C-H, ester C=O, acid, aldehyde or ketone C=O (weak), ether group, complex bond pattern below 1,500 cm^{-1}.
LC 7	Bonded OH, aliphatic C-H, ester C=O, conjugated C=C, possible ether group, miscellaneous $(CH_2)n$ groups.
LC 8	Bonded OH strong, aliphatic CH, strong C=C, ether group, some SiO_2 possible glycol ethers.

TABLE 46. IR REPORT
SAMPLE: PLANT V

LC 1	Aliphatic CH, $(CH_2)n$, where n > 4, hydrocarbons plus possible Si - CH_3.
LC 2	Aliphatic and aromatic CH, some ester C=0, silicones.
LC 3	Poor spectrum--low organic content? Aliphatic CH strong background adsorption.
LC 4	Bonded OH, aromatic and aliphatic CH, ester C=0, conjugated C=C, silicones.
LC 5	Identical to No. 4.
LC 6	Aliphatic CH, ester C=0, silicones, CH, various groups ester stronger, silicones weaker than in No. 4 or No. 5.
LC 7	Bonded OH, aliphatic CH, ester C=0, conjugated C=C ether group--possible glycol ether compounds.
LC 8	Strong bonded OH, weak aliphatic CH, ester C=0, strong C=C, some ether under SiO_2 adsorption.

TABLE 47. IR REPORT
SAMPLE: PLANT W

LC 1	Bonded OH, aliphatic CH, ester C=O, possible ether groups $(CH_2)n$ where $n > 4$. Not typical fraction 1.
LC 2	Aliphatic CH groups, ester C=O, weak spectrum.
LC 3	Aliphatic ester compounds, ester C=O, no aromatic CH.
LC 4	Trace OH, aliphatic CH, ester C=P, weak diffuse spectrum.
LC 5	Weak diffuse spectrum, poor background, OH (water?). Aliphatic CH, weak ester C=O, nonconjugated C=C.
LC 6	Bonded OH, aliphatic CH, ester C=O, long chain CH_2 groups, unsaturated acid, aldehyde or ketone, conjugated C=C, possible amide groups.
LC 7	Poor spectrum. Bonded OH, aliphatic CH, numerous C=O types, C=C, amide possible, very diffuse below 1,400 cm^{-1}.
LC 8	Weak spectrum, bonded OH--likely H_2O, very weak aliphatic CH, low organic content.

TABLE 48. IR REPORT
SAMPLE: PLANT X

LC 1	Aliphatic hydrocarbons, possible Si (CH_3).
LC 2	Aliphatic CH, ester C=0, conjugated C=C, hydrocarbon.
LC 3	Bonded OH, aliphatic CH, ester C=0, phthalate plus other types of ester materials.
LC 4	Similar to fraction No. 3.
LC 5	Bonded OH, trace aromatic CH, aliphatic CH, ester C=0, acid, aldehyde or ketone C=0, conjugated C=C, various straight and branched CH_2 chains.
LC 6	Bonded OH, aliphatic CH, ester C=0, conjugated and aromatic C=C, ether group, long CH_2 chains.
LC 7	Very similar to No. 6.
LC 8	Strong OH adsorption, similar to No. 6 and No. 7 but more OH and presence of SiO_2.

TABLE 49. LRMS REPORT
SAMPLE: PLANT A

1. Categories Present

LC	Intensity	Category	MW range
1	*	*	-
2	100	Aliphatics[†]	-
	100	Aromatics	-
3	100	Aliphatics	-
	100	Aromatics	-
4	*	*	-
5	*	*	-
6	100	Aliphatics	-
	10	Alcohols/ethers	-
	10	Phenols	-
	10	Esters	-
7	*	*	-
8	10	Aliphatics	-
	100	Alcohols/ethers	-

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	*	*	-	-
2	100	Parafinic/olefinic (or parafinic)[‡§]	-	-
	100	Alkyl benzenes, (91, 105, 114, 133 ions present)[cd]	-	-
3	100	(Same as LC-2)	-	-
4	*	*	-	-
5	*	*	-	-

See footnotes at end of table.

TABLE 49 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
6	100	Parafinic/olefinic (or cyclic parafinic)[†§]	-	-
	10	Alcoholic ethers (45 ions to 89 ions)[‡§]	-	-
	10	Bis(hydroxy-t-butyl phenyl) propane	340	$C_{23}H_{32}O_2$
	10	Di-n-octyl phthalate	390	$C_{24}H_{38}O_4$
7	*	*	-	-
8	10	Parafinic/olefinic (or cyclic-parafinic)[†§]	-	-
	100	Alcoholic-ethers (45 ions to 89 ions)[‡§]	-	-
		Ethers: Di-n-octyl phthalate	390	$C_{24}H_{38}O_4$

3. Other

LC	Intensity
1	*
2	No masses above 498
3	10: 368(100), 369(45), 353(20) 1: 345(100), 396(35), 411(30)
4	*
5	*
6	No masses above 414
7	*
8	No masses above 368 100; 254(100), 126(20), 127(15)

See footnotes at end of table.

TABLE 49 (con.)

Footnotes

*Organic weight of fraction below gravimetric threshold of 0.1 mg; therefore, no analysis was performed.

†Generally all ions up through 498 present in aliphatic-type pattern; however, all mass >100 are abnormally strong for typical aliphatics.

‡No molecular weight range determination possible.

§No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 50. LRMS REPORT
SAMPLE: PLANT B

1. Categories Present

LC	Intensity	Category	MW range
1	*	*	-
2	100	Aliphatics	-
	100	Esters	-
3	*	*	-
4	10	Aliphatic	-
5	*	*	-
6	1	Aliphatics	-
	100	Phenols	-
7	*	*	-
8	10	Aliphatics	-
	10	Alcohols/ethers	-
	10	Esters	-

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	*	*	-	-
2	100	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
3	*	*	-	-
4	10	Same as Plant A, Fraction 2	-	-
5	*	*	-	-
6	1	Same as Plant A, Fraction 2	--	-
	100	Bis(hydroxy-t-butyl phenol) propane	390	$C_{23}H_{32}O_2$
7	*	*	-	-

See footnotes at end of table.

TABLE 50 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
8	10	Same as Plant A, Fraction 2	-	-
	10	Alcohols/ethers (45 ions to 89 ions)†‡	-	-
	10	Same as esters, Plant A, Fraction 6	-	-

3. Other

LC	Intensity
1	*
2	No masses above 354
3	*
4	No masses above 483 10: 279(100), 294(28), 280(12) 100: 361(100), 356(36), 342(27) 100: 381(100), 396(15), 382(11) 100: 410(100), 151(37), 411(31) 100: 429(100), 444(23), 445(15)
5	*
6	No masses above 437
7	*
8	No masses above 414 100: 294(100), 127(20), 128(12)

---

[*] Organic weight of fraction below gravimetric threshold of 0.1 mg; therefore, no analysis was performed.

[†] No molecular weight range determination possible.

[‡] No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 51. LRMS REPORT
SAMPLE: PLANT F

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	-
2	100	Aliphatics	-
	100	Esters	-
3	1	Aliphatics	-
	100	Aromatics	-
	10	Esters	-
4	100	Aliphatics	-
	10	Aromatics	-
	10	Esters	-
5	10	Aliphatics	-
	1	Aromatics	-
	10/100	Phenols	-
	100	Esters	-
6	1	Aromatics	-
	10/100	Phenols	-
	10	Esters	-
7	100	Aliphatic	-
	10	Phenols	-
	100	Esters	-
8	10	Aliphatics	-
	100	Esters	-

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Primarily parafinic*†	-	-
2	100	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6	-	-

See footnotes at end of table.

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
3	1	Aliphatics*†	-	-
	100	Alkyl benzenes: Tri-t-butyl benzene	246	$C_{18}H_{3\underline{0}}$
	10	Same as esters, Plant A, Fraction 6	-	-
4	100	Same as Plant A, Fraction 2	-	-
	10	Tri-t-butyl benzene	246	$C_{18}H_{3\underline{0}}$
	10	Same as esters, Plant A, Fraction 6	-	-
5	10	Same as Plant A, Fraction 2	-	-
	1	Alkyl benzenes: Tri-t-butyl benzene*†	246	$C_{18}H_{3}0$
	10	Alkyl phenols (135, 107, 121, 148 ions)*†	-	-
	100	Bis (hydroxy-t-butyl phenyl) propane	390	$C_{23}H_{32}O_2$
	100	Same as esters, Plant A, Fraction 6	-	-
6	1	Tri-t-butyl benzene	246	$C_{18}H_{30}$
	10	Alkyl phenols (135, 107, 121, 149 ions)*†	-	-
	100	Bis (hydroxy-t-butyl phenyl) propane	390	$C_{23}H_{32}O_2$
	10	Same as esters, Plant A, Fraction 6	-	-
7	100	Primarily parafinic*†	-	-
	10	Bis (hydroxy-t-butyl) propane	390	$C_{23}H_{32}O_2$
	100	Same as esters, Plant A, Fraction 6	-	-
8	10	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6	-	-

See footnotes at end of table.

TABLE 51 (con.)

3. Other

LC	Intensity
1	No masses above 446
2	No masses above 354
3	No masses above 354
4	No masses above [illegible]
5	No masses above 340 10: 239(100), 240(20), 254(25)
6	No masses above 354 100: 45(100), 42(90) (2 compounds) - 100: 59(100) (4 compounds)
7	No masses above 354
8	No masses above 354

---

[*] No molecular weight range determination possible.

[†] No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 52. LRMS REPORT
SAMPLE: PLANT G

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	-
2	1	Aliphatics	-
	100	Esters	-
3	100	Aliphatics	-
	1	Esters	-
4	100	Aliphatics	-
	1	Esters	-
5	10	Aliphatics	-
	1	Phenols	-
	100	Esters	-
6	100	Aliphatics	-
	100	Alcohols/esters	-
	1	Phenols	-
	1	Esters	-
7	100	Aliphatics	-
	100	Alcohols/ethers	-
	1	Phenols	-
	1	Esters	-
8	100	Aliphatics	-
	10	Alcohols/ethers	-
	1	Esters	-

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Same as Plant F, Fraction 2	-	-
	1	Same as Plant A, Fraction 2	-	-
2	100	Same as esters, Plant A, Fraction 6	-	-

TABLE 52 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
3	100	Same as Plant A, Fraction 2	-	-
	1	Same as esters, Plant A, Fraction 6	-	-
4	100	Same as Plant A, Fraction 2	-	-
	1	Same as esters, Plant A, Fraction 6	-	-
5	10	Same as Plant A, Fraction 2	-	-
	1	Same as phenols, Plant A, Fraction 6	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
6	100	Same as Plant A, Fraction 2	-	-
	100	Alcoholic ethers with 41, 43, 45,	-	-
		55, 57, 59 ions*†	-	-
	1	Same as phenols, Plant A, Fraction 6	-	-
	1	Same as esters, Plant A, Fraction 6	-	-
7	100	Same as Plant A, Fraction 2	-	-
	100	Same as Fraction 6	-	-
	1	Same as phenols, Plant A, Fraction 6	-	-
	1	Same as esters, Plant A, Fraction 6	-	-
8	100	Same as Plant A, Fraction 2	-	-
	10	Same as Fraction 6	-	-
	1	Same as esters, Plant A, Fraction 6	-	-

See footnotes at end of table.

TABLE 52 (con.)

3. Other

LC	Intensity
1	No masses above 410
2	-
3	No masses above 378
4	No masses above 381
5	No masses above 325
6	No masses above 354 1:69(100), 41(87), 43(78)
7	No masses above 354 10:69 (100), 41(80), 43(78)
8	No masses above 354

[a] No molecular weight range determination possible.

[b] No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 53. LRMS REPORT
SAMPLE: PLANT L

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	–
2	100	Aliphatics	–
	100	Aromatics	–
3	100	Phenols	–
	100	Esters	–
4	100	Aliphatics	–
	100	Aromatics	–
	100	Phenols	–
	100	Esters	–
5	100	Aliphatics	–
	100	Phenols	–
	100	Esters	–
6	100	Phenols	–
	100	Esters	–
7	100	Aliphatics	–
	100/10	Phenols	–
	100	Esters	–
8	100	Aliphatics	–
	100	Esters	–

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Same as Plant F, Fraction 1	–	–
2	100	Same as Plant F, Fraction 1	–	–
	100	Same as Plant A, Fraction 3	–	–

TABLE 53 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
3	100 100	Alkyl phenols (135, 107, 121, 149 ions)*†	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
4	100	Same as Plant F, Fraction 1	-	-
	100	Same as Plant F, Fraction 3	-	-
	100	Di-t-butyl phenol	206	$C_{14}H_{22}O$
	100	Same as esters, Plant A, Fraction 6	-	-
5	100	Same as Plant F, Fraction 1	-	-
	100	Alkyl phenol (135, 107, 121, 149 ions)*†	- -	- -
	100	Same as esters, Plant A, Fraction 6	-	-
6	100	Alkyl phenols (135, 107, 121, 149 ions)*†	-	-
	100	Phthalate, probably di-C_8 alkyl but with a new series of ions added (203, 237, 251, 265, 279)	-	-
7	100	Same as Plant A, Fraction 2	-	-
	100	Alkyl phenols (135, 107, 121, 144 ions)*†	-	-
	10	Bis-(hydroxy-t-butyl phenyl) propane	340	$C_{23}H_{32}O_2$
8	100	Same as Plant A, Fraction 2	-	-
	100	Same as Plant A, Fraction 6	-	-

See footnotes at end of table.

TABLE 53 (con.)

3. Other

LC	Intensity
1	No masses above 367
2	No masses above 367
3	No masses above 10: 69(100), 41(80), 43(78)
4	No masses above 429
5	No masses above 429 10: 69(100), 41(80), 43(78)
6	No masses above 340
7	No masses above 381 100: 69(100), 41(80), 43(78) 1: 158 1: 200(100), 201(86) 10: 280(100), 279(98)
8	No masses above 279 1: Cesium iodide from infrared plates

---

*No molecular weight range determination possible.

†No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 54. LRMS REPORT
SAMPLE: PLANT N

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	-
	10	Esters	-
2	100	Aliphatics	-
	100	Aromatics	-
	100	Esters	-
[illegible]	[illegible]	Aliphatics	-
	100	Aromatics	-
	10	Esters	-
4	100	Aliphatics	-
	10	Esters	-
5	100	Aliphatics	-
	10	Phenols	-
	100	Esters	-
6	100	Aliphatics	-
	10	Aromatics	-
	10	Amines	-
	10/10	Phenols	-
	10	Esters	-
7	100	Aliphatics	-
	10	Phenols	-
	1	Phenols	-
	1	Esters	-
8	100	Aliphatics	-
	10	Phenols	-
	100	Esters	-

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Same as Plant A, Fraction 2	-	-
	10	Same as esters, Plant A, Fraction 6	-	-

TABLE 54 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
2	100	Same as Plant A, Fraction 2	-	-
	100	Alkyl benzene (91, 105, 119 ions)*†	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
3	100	Same as Plant A, Fraction 2	-	-
	100	Same as Plant A, Fraction 3	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
4	100	Same as Plant A, Fraction 2	-	-
	10	Same as Plant A, Fraction 6	-	-
5	100	Same as Plant A, Fraction 2	-	-
	10	Same as Plant F, Fraction 5	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
6	100	Same as Plant A, Fraction 2	-	-
		t-Butyl dichlorobenzene	202	$C_{10}H_{12}Cl_2$
	10	Dichloroaniline	161	$C_6H_5NCl_2$
	10	Same as phenols, Plant F, Fraction 5	-	-
	10	Same as phenols, Plant A, Fraction 6	-	-
	10	Phthalate, probably di-C_8 alkyl but with a new series of ions added: 223, 237, 251, 265, 279	- 390	- $C_{24}H_{38}O_4$
7	100	Same as Plant A, Fraction 2	-	-
	10	Same as phenols, Plant F, Fraction 5	-	-
	1	Same as phenols, Plant A, Fraction 6	-	-
	1	Same as esters, Plant A, Fraction 6	-	-
8	100	Same as Plant A, Fraction 2	-	-
	10	Same as phenols, Plant F, Fraction 5	-	-
	100	Same as esters, Plant A, Fraction 6	-	-

See footnotes at end of table.

TABLE 54 (con.)

3. Other

LC	Intensity
1	No masses above 428
2	No masses above 394
3	No masses above 498 10: 368(100), 369(30), 353(20) 10: 395(100), 396(30) 1: 410(100), 411(30)
4	No masses above 499 10: 368(100), 369(30), 353(20) 10: 395(100), 396(30) 10: 410(100), 411(30)
5	No masses above 279
6	No masses above 490 1: 155
7	No masses above 340
8	No masses above 279 1: Cesium iodide

---

[*] No molecular weight range determination possible.

[†] No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 55. LRMS REPORT
SAMPLE: PLANT S

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	-
2	100	Aliphatics	-
	100	Aromatics	-
3	100	Aliphatics	-
	10	Aromatics	-
	1	Esters	-
4	10	Aliphatics	-
	100	Aromatics	-
	10	Aromatics	-
	10	Phenols	-
	10	Phenols	-
5	100	Aliphatics	-
	100	Aromatics	-
6	1	Aliphatics	-
	100	Aromatics	-
	10	Phenols	-
	10	Esters	-
7	1	Aliphatics	-
	10	Aromatics	-
	100	Esters	-
8	100	Aliphatics	-
	10	Esters	-

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Same as Plant A, Fraction 2	-	-

TABLE 55 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
2	100	Same as Plant A, Fraction 2	-	-
	100	Same as Plant A, Fraction 2	-	-
3	100	Same as Plant A, Fraction 2	-	-
	10	Same as Plant F, Fraction 3	-	-
	1	Same as Plant A, Fraction 6	-	-
4	10	Same as Plant A, Fraction 2	-	-
	100	Toluene-sulfonyl groups (91, 155 ions)	- -	- -
	10	Best identity is p-toluene sulfonamide	-	-
	10	Same as Plant F, Fraction 3	-	-
	10	Di-t-butyl phenol	206	$C_{14}H_{22}O$
5	100	Same as Plant A, Fraction 2	-	-
	100	Toluene-sulfonyl group (91, 155 ions)	-	-
		Best identity p-toluene sulfonamide		
6	1	Same as Plant A, Fraction 2	-	-
	100	Toluene-sulfonyl group (91, 155 ions)*†	-	-
	10	Same as phenols, Plant A, Fraction 6	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
7	1	Same as Plant A, Fraction 2	-	-
	10	Same as aromatics, Plant A, Fraction Fraction 4	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
8	100	Same as Plant A, Fraction 2	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
	1	Methyl esters (74, 89 ions)*†	-	-

See footnotes at end of table.

TABLE 55 (con.)

3. Other

LC	Intensity
1	No masses above 446
2	No masses above 446
3	No masses above 452
4	No masses above 477 1: 381(100), 382(27), 396(17)
5	No masses above 354 100: 98(100), 97(75)
6	No masses above 446 10: 90(100), 91(68), 106(58)
7	No masses above 446 10: 90(100), 91(68), 106(58)
8	No masses above 354

---

[*] No molecular weight range determination possible.

[†] No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 56. LRMS REPORT
SAMPLE: PLANT T

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	-
	1	Esters	-
2	100	Aliphatics	-
	100	Aromatics	-
	10	Esters	-
3	100	Aliphatics	-
	100	Aliphatics	-
	10	Aromatics	-
	10	Esters	-
4	100	Aliphatics	-
	10	Aromatics	-
	10	Phenols	-
	1	Esters	-
5	10	Aliphatics	-
	100	Esters	-
6	100	Aliphatics	-
	100	Phenols	-
	100	Phenols	-
	100	Esters	-
7	100	Aliphatics	-
	100	Phenols	-
	100	Esters	-
8	10	Aliphatics	-
	10	Esters	-

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 56 (con.)

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Same as Plant A, Fraction 2	-	-
	1	Same as esters, Plant A, Fraction 6	-	-
2	100	Same as Plant A, Fraction 2	-	-
	100	Same as Plant A, Fraction 2	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
3	100	Same as Plant A, Fraction 2	-	-
	100	Same as Plant F, Fraction 1	-	-
	10	Same as Plant F, Fraction 3	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
4	100	Same as Plant F, Fraction 1	-	-
	10	Same as Plant F, Fraction 3	-	-
	10	Di-t-butyl phenol	206	$C_{14}H_{22}O$
	1	Same as Plant A, Fraction 6	-	
5	10	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
6	100	Same as Plant F, Fraction 1	-	-
	100	Same as Plant F, Fraction 3	-	-
	100	Same as phenols, Plant A, Fraction 6	-	-
	100	Same as esters, Plant A, Fraction 6	-	-

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent

TABLE 56 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
7	100	Same as Plant A, Fraction 2	-	-
	100	Same as phenols, Plant F, Fraction 5	-	-
	100	Phthalate, probably di-C_8 alkyl but with a new series of ions added: 223, 237, 251, 265, 279 ions	390	$C_{24}H_{38}O_4$
8	10	Same as Plant A, Fraction 2	-	-
	10	Same as esters, Plant A, Fraction 6.	-	-

3. Other

LC	Intensity
1	No masses above 362
2	No masses above 312 10:69 (100), 41(80), 43(78)
3	No masses above 486 1:314(100), 315(25) 10:410(100), 411(32)
4	No masses above 396 10:69(100), 41(80), 43(78)
5	No masses above 396
6	No masses above 340 100:99

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 56 (con.)

3. Other (con.)

LC	Intensity
7	No masses above 279
8	No masses above 336 100:117(100), 59(44) 10:69(100), 41(80), 43(78) Also many organo-silicon ions: e.g., 207, 221, etc.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent

TABLE 57. LRMS REPORT
SAMPLE: PLANT U

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	-
2	100	Aliphatics	-
	100	Aromatics	-
3	100	Aliphatics	-
4	100	Aliphatics	-
	100	Esters	-
	10	Esters	-
	10	Amines	-
5	100	Aliphatics	-
	100	Esters	-
	10	Amines	-
6	100	Aliphatics	-
	100	Esters	-
	10	Amines	-
7	100	Aliphatics	-
	100	Phenols	-
	10	Esters	-
	100	Esters	-
8	100	Aliphatics	-
	10	Esters	-

NOTE: LRMS performed on CH_2Cl_2 extract of second effluent.

TABLE 57 (con.)

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Same as Plant F, Fraction 1	-	-
2	100	Same as Plant A, Fraction 2	-	-
	100	Same as Plant A, Fraction 2	-	-
3	100	Same as Plant A, Fraction 2	-	-
4	100	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
	10	Vinyl stearate	310	$C_{20}H_{38}O_2$
	10	Azo compound: Naphthalene-mno-azo-benzene	248	$C_{16}H_{12}ON_2$
5	100	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
	10	Halogenated amines: chloroaniline	127	C_6H_6Cl
6	100	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6 and vinyl stearate	310	$C_{20}H_{38}O_2$
	10	Chloroaniline	127	C_6H_6Cl
7	100	Same as Plant A, Fraction 2	-	-
	100	Same as phenols, Plant A, Fraction 6	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
	100	Vinyl stearate	310	$C_{20}H_{38}O_2$

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 57 (con.)

2. Subcategories, Specific Compounds (con.)

LC	Intensity	Compound	m/e	Composition
8	100	Same as Plant A, Fraction 2	-	-
	10	Same as esters, Plant A, Fraction 6	-	-

3. Other

LC	Intensity
1.	No masses above 404
2	No masses above 410
3	No masses above 398 in aliphatic type pattern; all masses above 100 are abnormally strong for typical aliphatics.
4	No masses above 411 10:410(100), 411(30)
5	No masses above 444 10:429(100), 444(20)
6	No masses above 496
7	No masses above 495 Similar to unusual pattern in Fraction 3 through mass 495.
8	No masses above 340 100:155 10:254, 127(diatomic iodine or probably napthyl iodide). 1: Cesium iodide.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 58. LRMS REPORT
SAMPLE: PLANT V

1. Categories Present

LC	Intensity	Category	MW Range
1	100	Aliphatics	-
	10	Esters	-
2	100	Organo-silicon species	-
	10	Esters	-
3	100	Aliphatics	-
	1	Aromatics	-
	100	Esters	-
4	10	Aliphatics	-
	10	Organo-silicon species	-
	100	Esters	-
5	10	Phenols	-
	100	Esters	-
6	100	Aliphatics	-
	100	Phenols	-
	100	Esters	-
7	10	Aliphatics	-
	100	Alcohols/ethers	-
	100	Esters	-
8	100	Esters	-

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 58 (con.)

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	Same as Plant F, Fraction 1	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
2	100	73 (dominant), 147, 207, 221, 355 ions*†	-	-
	10	Same as esters, Plant A, Fraction 6	-	-
3	100	Same as Plant A, Fraction 2	-	-
	1	Same as Plant A, Fraction 2	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
4	10	Same as Plant F, Fraction 1	-	-
	10	73(dominant), 147 ions*†	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
5	10	Same as phenols, Plant A, Fraction 6	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
6	100	Same as Plant F, Fraction 1	-	-
	100	Same as phenols, Plant A, Fraction 6	-	-
	100	Same as esters, Plant A, Fraction 6	-	-
7	10	Same as Plant F, Fraction 1	-	-
	100	Pattern indicates alcoholic ethers 41, 43, 49(dominant) and 55, 57, 59 (dominant) ion clusters*†	-	-
8	100	Same as esters, Plant A, Fraction 6	-	-
	100	Same as esters, Plant A, Fraction 6	-	-

*No molecular weight range determination possible.
†No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 58 (con.)

3. Other

LC	Intensity
1	No masses above 451
2	No masses above 491
3	No masses above 296 10L69(100), 41(80), 43(78)
4	No masses above 477 100:69(100), 41(80), 43(78)
5	No masses above 340 10:69(100), 41(80), 43(78)
6	No masses above 253 10:156(100), 155(35) Possibly bipyridyl or phenyl cyclohexadiene MW 156 each
7	No masses above 373
8	No masses above 279

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 59. LRMS REPORT
SAMPLE: PLANT W

1. Categories Present

LC	Intensity	Category	MW range
1	100	Aliphatics	-
	100	Aliphatics	-
	1	Esters	-
2	100	Aliphatics	-
	10	Aromatics	-
	100	Esters	-
3	10	Aliphatics	-
	1	Esters	-
4	100	Aliphatics	-
	10	Esters	-
5	100	Aliphatics	-
	10	Phenol	-
	10	Esters	-
6	100	Aliphatics	-
	100	Phenols	-
	10	Phenols	-
7	100	Aliphatics	-
	10	Esters	-
8	100	Aliphatics	-

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 59 (con.)

2. Subcategories, Specific Compounds

LC	Intensity	Compound	m/e	Composition
1	100	n-Paraffins*†	-	-
	100	Same as plant A, fraction 2	-	-
	1	Same as esters, plant A, fraction 6	-	-
2	100	Same as plant A, fraction 2	-	-
	10	Same as plant A, fraction 2	-	-
	100	Same as esters, plant A, fraction 6	-	-
3	10	Same as plant A, fraction 2	-	-
	1	Same as plant A, fraction 6	-	-
4	100	Same as plant A, fraction 2	-	-
	10	Same as esters, plant A, fraction 6	-	-
5	100	Same as plant A, fraction 2	-	-
	10	Same as phenols, plant A, fraction 6	-	-
	10	Same as esters, plant A, fraction 6	-	-
6	100	Same as plant A, fraction 2	-	-
	100	Same as plant F, fraction 5	-	-
	10	Same as phenols, plant A, fraction 6	-	-
	10	Same as esters, plant A, fraction 6	-	-
7	100	Same as plant A, fraction 2	-	-
	10	Same as esters, plant A, fraction 6	-	-
8	100	Same as plant A, fraction 2	-	-

*No molecular weight range determination possible.

†No composition determination possible.

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.

TABLE 59 (con.)

3. Other

LC	Intensity
1	No masses above 424 1:368(100), 369(45), 353(18)
2	No masses above 409 100:368(100), 369(45), 353(18)
3	No masses above 485 100:368(100), 369(45), 353(18)
4	No masses above 495 10:69(100), 41(80), 43(78) 100:368(100), 369(45), 353(18)
5	No masses above 480 100:368(100), 369(45), 353(18)
6	No masses above 451
7	No masses above 495 10:69(100), 41(80), 43(78) 100:384(100), 383(98), 368(90), 382(68)
8	No masses above 499 10:Cesium iodide or nathyl iodide 100:69(100), 41(80), 43(78) 10:383(100), 368(96), 382(72), 369(64)

NOTE: LRMS performed on CH_2Cl_2 extract of secondary effluent.